

**ATTACHMENT A**

**STANDARD OPERATING PROCEDURES**

<b>SOP Description</b>	<b>SOP ID</b>
Surface Water Sampling	No. 3 (Rev. 0)
Water Sampling With Syringe Filters	No. 3A (Rev. 0)
Equipment Decontamination	No. 7 (Rev. 0)
Sample Handling and Shipping	No. 8 (Rev. 0)
Field Documentation	No. 9 (Rev. 6)
Field Equipment Calibration	No. 10 (Rev. 1)
GPS Data Collection	No. 11 (Rev. 1)
Investigation Derived Waste (IDW) Management	No. 12 (Rev. 0)
Automated Water Sampling	No. 14 (Rev. 0)
Stream Pool Classification and Characterization	No. 19 (Rev. 0)
Libby OU3 SW Temperature Continuous Monitoring	No. 20 (Rev. 0)

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007

OU3 SOP 3 (Rev. 0)

Title: SURFACE WATER SAMPLING

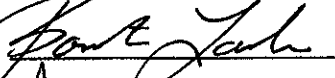

**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

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EPA Remedial Project Manager

9/26/07  
9/26/07

SOP Author

Revision Number	Date	Reason for Revision
0	09/26/2007	-

## 1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-12, Surface Water Sampling, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. This SOP describes methods and equipment commonly used for collecting environmental samples of surface water for either on-site examination and chemical testing or for laboratory analysis. The information presented in this SOP is generally applicable to all environmental sampling of surface waters except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

This document focuses on methods and equipment that are readily available and typically applied in collecting surface water samples. It is not intended to provide an all-inclusive discussion of sample collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in surface water sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## 3.0 DEFINITIONS

**Bailer:** A long, narrow, tubular device with an open top and a check valve at the bottom. Bailers may be made of Teflon<sup>®</sup>, Polyethylene, or stainless steel.

**Specific Conductance:** How well water can conduct an electrical current.

**Dip Sampler:** A sample collection container that may be held directly or attached to a pole, used to collect surface water samples from the surface or just beneath the surface of a water body.

**Dissolved Oxygen (DO):** A measure of the quantity of oxygen dissolved in water. DO data is collected in the field using direct measure probes.

**Environmental Sample:** A liquid sample collected for chemical analysis. These samples are used to support remedial investigation, feasibility studies, treatability studies, remediation design and performance assessment, waste characterization, etc.

**Oxidation-Reduction Potential (ORP):** A measurement of the reducing or oxidation potential of a given system or medium. ORP data is collected in the field using direct-measure probes.

**Peristaltic Pump:** A low volume pump that operates by suction lift.

**pH:** A measure of the acidity or alkalinity of a solution, numerically equal to 7 for neutral solutions, increasing with increasing alkalinity to a maximum value of 14, and decreasing with increasing acidity to a minimum value of 1.

**Temperature:** A measure of the thermal energy contained in a given system. Units are commonly in degrees Celsius (°C).

**Turbidity:** Cloudiness in water due to suspended and colloidal organic and inorganic material. Units are commonly in Nephelometric Turbidity Units (NTUs).

#### **4.0 RESPONSIBILITIES**

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be

involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Selects site-specific field sampling program with input from other key project staff, and applicable oversight agencies.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer:** Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

**Sampling Technician (or other designated personnel):** Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

## 5.0 SURFACE WATER SAMPLING PROCEDURES

### 5.1 Background

The methods and procedures described in this SOP were developed from these sources:

U.S. Geological Survey, variously dated. *National field manual for the collection of water-quality data: U.S. Geological Survey Techniques of Water-Resources Investigations, Book 9, Chapters A1-A9*. Available online at <http://pubs.water.usgs.gov/twri9A>.

- U.S. Environmental Protection Agency, Region 8, 2003. *SOP EH-#1 Technical Standards Operating Procedure – Surface Water Sampling East Helena Site, Montana*. Available online at

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[http://www.epa.gov/region8/r8risk/pdf/r8-src\\_eh-01.pdf](http://www.epa.gov/region8/r8risk/pdf/r8-src_eh-01.pdf)

## 5.2 Surface Water Sample Collection

**Rivers, Streams and Ponds:** Where multiple sampling stations exist along a moving water source (i.e., a creek or drainage channel), water samples generally will be collected from downstream to upstream locations, to minimize the effect of sampling activities on the samples collected. The samples will be grab samples collected from representative flowing water (usually the mid-channel), that is the portion of the water with the maximum flow at any given sampling station, unless otherwise specified.

A surface water sample will be collected according to one of the following, or similar, techniques.

1. Direct Method -- Sample bottle is uncapped and inverted, submerged to the specified depth, turned upright pointing upstream, removed from the water, and then capped. Add preservative, if any, after sample collection.
2. Dipper Method -- Sample bottle or container attached to a pole is dipped in the water, raised above the water, and then capped (if actual sample bottle used).
3. Bailer Method -- A appropriate sampling bailer with a ball check valve is submerged to the desired sample depth, either directly or by suspending the bailer on a rope from a pole.
4. Peristaltic Pump Method -- The sample is collected through a section of new, clean, flexible Tygon (polyvinylchloride) tubing. The tubing intake will be secured manually or by attaching weights. This procedure may be modified to collect the sample through a Teflon tube into a sample flask by running the pump on a vacuum.
5. Kemmerer Bottle -- Use a properly decontaminated Kemmerer bottle. Set the sampling device so that the upper and lower stoppers are pulled away from the body, allowing water to enter the tube. Lower the pre-set sampling device to the predetermined depth. Avoid disturbing the bottom. Once at the required depth, send the weighted messenger down the suspension line, closing the device. Retrieve the sampler and discharge the first 10-20 mL from the drain to clear water that may not be representative of the sample. Repeat as needed to fill collect the needed volume.
6. Van Dorn Sampler -- Set the device so that the end stoppers are pulled away from the body allowing surface water to enter the tube. Lower the sampler to the

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predetermined depth. Once at the required depth send the weighted messenger down the suspension line, closing the sampling device. Retrieve the sampler and decant the first 10-20 mL from the drain to clear water that may not be representative of the sample from the valve. Repeat as needed until the required volume to fill sample bottles is collected.

7. Bacon Bomb Sampler -- Lower the bacon bomb sampler carefully to the desired depth, allowing for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line until taught. This will allow the sampler to fill. Release the trigger line and retrieve the sampler. Decant the first 10-20 mL from the drain to clear water that may not be representative of the sample from the valve. Repeat as needed until the required volume to fill sample bottles is collected.

For very shallow waters, a syringe method can be used, where a disposable plastic filtering syringe may be used to collect a sample without disturbing the sediment. Alternatively, at low flowing seeps and springs a small depression may be created to capture water. If a depression is made, the disturbance-related turbidity should be allowed to clear (settle) before the sample is collected.

### **5.3 Sampling Equipment and Techniques**

The selection of sampling equipment listed above depends on the site conditions and sample type required. In addition, the following equipment is needed to collect surface water samples:

- Field notebook, indelible marker
- Global Positioning System (GPS) unit
- Marking stakes
- Digital Camera
- Compass
- 100 m measuring tapes
- Detergent solution (0.1-0.3 % Alconox)
- Distilled water
- Latex gloves
- Ziploc bags
- Paper Towel



- Chain of custody and sample labels
- Coolers
- Sample bottles
- Plastic sheeting

For collecting surface water samples, the procedures outlined below shall be followed.

1. Don appropriate health and safety equipment
2. Setup clean plastic sheeting in area for processing samples
3. Collect a surface water sample beginning from the most downstream location using one of the methods specified above
4. Place a stake or pole at or near the sampling location for future ease of identification
5. The first collected water will be used to rinse the sampling equipment. Sample bottles that do not contain preservative should be rinsed with the sample water prior to filling
6. Pour the sample from the sampling equipment down the side of the sample container in such a manner as to minimize turbulence during the transfer, or alternatively, collect the sample directly into the sample container. However, a primary concern with sample collection directly into sample containers is the loss of sample preservative from the sample container as it is dipped directly into the surface water.
7. Collect samples for Volatile Organic Compounds (VOC) first. Do not collect samples for VOC analysis using a peristaltic pump.
8. Label sample containers with the sample location and sample analysis information in accordance with the procedures in SOP-9.

9. Measure and record water quality parameters pH, DO, temperature, specific conductance, ORP, and turbidity using equipment calibrated according to manufacturer's specifications
10. Measure stream discharge according to SOP-4 and locate the sample using a site map or GPS according to SOP-11.
11. Pack samples containers to avoid leakage or breakage during shipment.
12. Store and ship samples on ice at 4 degrees Celsius. For further details on shipping and handling refer to SOP-8.

**5.4 Sample Filtration:** When required, a field-filtered water sample will be collected using a disposable, in-line 0.45 µm filter. The water sample will be pumped through the filter using a peristaltic pump and a section of Tygon (polyvinylchloride) or non-reactive (Teflon®) tubing or other appropriate method. An aliquot of approximately 100 ml of sample will be run through the tubing and filter prior to collection into the sampling containers. Both the filter and tubing will be disposed of between samples.

**5.5 Sample Containers and Volumes:** Certified clean sample containers appropriate to the analytical method will be obtained from the water analysis laboratory or other approved source. Different containers will be required for specific groups of analytes in accordance with U. S. EPA Methods, project specific requirements, and/or other local jurisdictional guidance. The sampler will confirm with the laboratory performing the analyses that the appropriate bottlenecks and preservatives are used and ensured that sufficient volume of the sample is collected.

Sample bottles and bottle caps will be protected from dust or other contamination between time of receipt by the sampling personnel and time of actual usage at the sampling site. Sampling equipment that will be used at multiple sampling locations will be cleaned after sampling at each location is completed. Decontamination of equipment will be completed in accordance with SOP-7.

**5.6 Sample Preservation and Storage:** If required by the project or analytical method, water samples submitted for chemical analysis will be stored at 4°C in ice-cooled, insulated containers immediately after collection. Preservation and storage methods depend on the chemical constituents to be analyzed and should be discussed with the water analysis laboratory prior to sample collection. EPA and/or other local jurisdictional requirements and/or the requirements of a project-specific plan (e.g., sampling and analysis plan, work plan, quality assurance project plan, etc.) shall be adhered to in preservation and storage of water samples.

**5.7 Documentation:** At each surface water station, sample details will be recorded on a Surface Water field sample data sheet (FSDS) form (see SOP-9, Attachment 1). Sampling conditions, including any deviations from this SOP in field logbook according to SOP-9. The sampling conditions that should be noted in the field log book will include:

- A. Project identification;
- B. Location identification (sampling station);
- C. Detailed description of sampling location;
- D. Sampling methods and equipment;
- E. Condition of water (standing or moving);
- F. Instrument calibration and cleaning record; and
- G. Sketch map showing location of sampling station and permanent landmarks, and locate using a global positioning system according to SOP-11.

When the sampling activity is completed, the record will be checked by the Project Manager or his/her designee, and the original record will be placed in the project file.

## **6.0 QUALITY ASSURANCE AND QUALITY CONTROL**

Field splits, field blanks, equipment rinsates, and matrix spike samples will be collected at the frequencies documented in the field sampling plan. Calibration checks will be performed at least once prior to and at least once following each day of instrument use in the field and the results documented in the field log book. All sampling data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

## **7.0 DECONTAMINATION**

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

## **8.0 REFERENCES**

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Feltz, H.R., 1980. Significance of Bottom Material Data in Evaluating Water Quality in Contaminants and Sediments. Ann Arbor, Michigan, Ann Arbor Science Publishers, Inc., Vol. 1, pp. 271-287.

Kittrell, F.W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135 pp.

U.S. Environmental Protection Agency (USEPA), 1980. Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

U.S. Environmental Protection Agency. 2007. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/600/B-07/001. April 2007.

U.S. Geological Survey (USGS), 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, Reston, Virginia.

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure
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Date: February 24, 2011

OU3 SOP 3A (Rev. 0)

Title: WATER SAMPLING WITH SYRINGE FILTERS

**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

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EPA Remedial Project Manager

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SOP Author

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Revision Number	Date	Reason for Revision
0	02/24/2011	--

## **1.0 INTRODUCTION**

This standard operating procedure (SOP) describes a method for collection and filtration of water samples for the analysis of free (un-bound) Libby Amphibole (LA) asbestos. This SOP is applicable to site water or laboratory water samples collected as part of the Remedial Investigation in Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site.

## **2.0 HEALTH AND SAFETY WARNING**

All personnel engaged in surface water sampling in OU3 must follow health and safety protocols described in the appropriate health and safety plan. Inhalation exposure to asbestos during sampling may increase the risk of lung cancer, mesothelioma, asbestosis, and other respiratory diseases.

## **3.0 RESPONSIBILITIES**

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Selects site-specific field sampling program with input from other key project staff, and applicable oversight agencies.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer:** Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

**Sampling Technician (or other designated personnel):** Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).



## **4.0 SYRINGE FILTRATION PROCEDURE**

### **4.1 Background**

Measurement of LA in site or laboratory waters is complicated by the finding that if the water is not completely sterile, organic matter associated with microbial contamination tends to form, which tends to cause the fibers to clump together (EPA 1983a). This causes two effects: a) asbestos fibers that are clumped together are difficult to observe and count using transmission electron microscopy (TEM), and this may lead to a decrease in estimated concentration of LA in the water, and b) fibers within clumps of organic matter tend to adhere to the walls of the sample bottles, thus decreasing the concentration of fibers in the water. The magnitude of these effects is time-variable, and depends on the amount of organic matter present and the time the sample is held before filtering. Both phenomena (fiber clumping, fiber adherence to container walls) have been observed in studies performed to date by EPA at the Libby OU3 site, including a juvenile rainbow trout toxicity test performed using site waters in 2009, and analysis of surface water samples collected at stream sampling station LRC-06 in July 2009.

EPA developed Analytical Method 100.1 (EPA 1983b) for the analysis of total asbestos in water. This method involves treating the water sample with ozone, ultraviolet light, and sonication before filtration. This treatment oxidizes organic material that is present in the water or on the walls of the sample bottle, destroying the material that causes clumping and binding of fibers. Based on studies performed by EPA, this treatment allows good recovery of fibers under a variety of starting conditions.

In some cases, it may also be valuable to measure the concentration of free asbestos in water samples, where “free asbestos” refers to asbestos fibers that exist in the water but are not associated with clumps of organic material.

When seeking to measure the concentration of free asbestos in water, the details of how the water is handled before filtration may be important. For example, if the water sample is placed in a bottle for transport to the laboratory for filtration, it is possible that additional clumping might occur and/or that binding of clumps to the bottle wall might occur before filtration occurs. If so, this could yield results that are not representative of the true concentration of free asbestos in the water at the time of sampling.

One way to avoid this potential problem is to filter the water sample directly at the site of collection, and never place it in a bottle for transport to the laboratory. The purpose of this SOP is to describe a method for the preparation of water filters using a syringe filter technique.

## **4.2 Surface Water Syringe Sample Collection**

### **4.2.1 Sampling Equipment**

The key equipment needed to collect water syringe filter samples is:

- Plastic syringes, with luerlok fitting, adequate to hold the volume of water specified in the project-specific SAP (typically 10-50 mL)
- Filter holders for 25 mm diameter filter (e.g., Millipore Swinnex SX0002500, or equivalent)
- 25 mm diameter polycarbonate filters with 0.1 um pore size

In addition, the following equipment is needed to support the documentation of sampling locations, and for sample handling after collection:

- Field notebook, indelible marker
- Global Positioning System (GPS) unit
- Digital Camera
- Ziploc bags
- Chain of custody and sample labels
- Coolers

### **4.2.2 Sampling Protocol**

Where multiple sampling stations exist along a moving water source (i.e., a creek or drainage channel), water samples generally will be collected from downstream to upstream locations, to minimize the effect of sampling activities on the samples collected.

For collecting syringe filter samples, the procedure outlined below shall be followed.

1. Don appropriate health and safety equipment
2. Fill a syringe with water from the test location
3. Turn the syringe upward, and tap to cause any air bubbles to rise to the open end
4. Push the syringe plunger upward, expelling all air bubbles, and adjusting the volume in the syringe to the volume specified in the project-specific SAP
5. Attach a filter holder containing a 25 mm polycarbonate filter with 0.1 um pore size.
6. Turn the syringe with filter holder downward, and hold as nearly vertical as possible.
7. Press the syringe plunger downward, forcing all of the water in the syringe the water through the filter.
8. Detach the filter holder from the syringe. Reattach a clean 10 mL plastic syringe, with the plunger withdrawn to the 10 mL mark. Holding vertically, press the plunger

- down, forcing 10 mL of air through the filter and filter holder. This will expel any loose water droplets from the filter holder.
9. Detach the filter holder and place in a Ziploc baggie, clearly labeled with a unique sample identification number and sample information, as specified in the project-specific SAP and in accordance with the procedures in SOP No. 9.
  10. Pack the Ziploc baggie in a cooler at 4 degrees Celsius for safe storage during remaining field activities.
  11. Transport the cooler to the analytical laboratory for further processing of the syringe filters every day that samples are collected. Do not hold overnight before delivery.

#### **4.3 Documentation**

At each surface water station, sample details will be recorded on a Surface Water field sample data sheet (FSDS) form (see SOP No. 9, Attachment 1).

In addition, a field logbook will be maintained in according to SOP No. 9. Data items that shall be recorded in the field logbook include:

- a. Project identification (e.g., OU3 RI Phase IVB)
- b. Location identification (sampling station), including global positioning system coordinates (see SOP No. 11)
- c. Date and time of sample collection
- d. Any deviations for this SOP, and any field conditions that may influence sample quality or relevance

When the sampling activity is completed, the record will be checked by the Project Manager or his/her designee, and the original record will be placed in the project file.

#### **5.0 QUALITY ASSURANCE AND QUALITY CONTROL**

Field blank and field duplicate samples will be collected at the frequencies documented in the field sampling plan.

#### **6.0 DECONTAMINATION**

All syringes and filter holders used in the sampling process shall either be new (un-used) or else shall be decontaminated prior to field use and between sample locations.

#### **7.0 REFERENCES**

EPA. 1983a. Development of Improved Analytical Techniques for Determination of Asbestos in Water samples. Report prepared for the U.S. Environmental Protection Agency,

Environmental Research Laboratory, Office of Research and Development, Athens, GA, by the Ontario Research Foundation, Mississauga, Ontario. EPA-600/4-83-042. September, 1983.

EPA. 1983b. Analytical Method for Determination of Asbestos in Water. U.S. Environmental Protection Agency, Environmental Research Laboratory, Office of Research and Development, Athens GA. EPA-600/4-83-043. September, 1983.

Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007

OU3 SOP 7 (Rev. 0)

Title: EQUIPMENT DECONTAMINATION

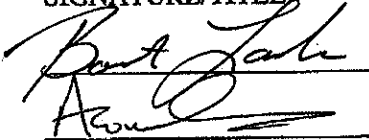
APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager



9/26/07

SOP Author



9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--

## 1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is based on MWH SOP-02, Equipment Decontamination, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. Decontamination of drilling, sampling, and monitoring equipment is a necessary and critical aspect of environmental field investigations. Proper decontamination is a key element in reducing the potential for cross-contamination between samples from different locations, as well as ensuring that samples are representative of the sampled materials. Improper decontamination may result in costly re-collection and re-analysis of samples. All equipment used in the sampling process will be properly decontaminated prior to the collection of each sample and after completion of sampling activities.

The procedures outlined in this SOP will be followed during decontamination of field equipment used in the sampling process, including drilling, soil/water sample collection, and monitoring activities. Any deviations from these procedures will be noted in the field notebooks and approved by the appropriate oversight agency, if significant. Three major categories of field equipment, along with applicable decontamination methods for each, are discussed below.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in equipment decontamination must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## 3.0 DEFINITIONS

**Bailer:** A cylindrical tool designed to remove material from a well. A valve at the bottom of the bailer retains the contents in the bailer.

**Bladder Pump:** Groundwater sampling equipment consisting of a flexible bladder, usually made of Teflon<sup>®</sup>, contained within a rigid cylindrical body (commonly made of stainless steel). The lower end of the bladder is connected to the intake port through a check valve, while the upper end is connected through a second check valve to a sampling line that leads to the ground surface.

**Brass Sleeve:** Hollow, cylindrical sleeves made of brass and used as liners in split-spoon samplers for collection of undisturbed samples.

**Auger Flight:** An individual auger section, usually 5 feet in length.

**Continuous Core Barrel:** 3-5 foot long steel barrels that can be joined together to allow continuous cores to be collected during a single run.

**Drill Pipe:** Hollow metal pipe used for drilling, through which soil and groundwater sampling devices can be advanced for sample collection.

**Peristaltic Pump:** A low-volume suction pump. The compression of a flexible tube by a rotor results in the development of suction.

**Source Water:** A drilling quality water source identified to be used for steam cleaning. This source should be sampled at the beginning of each field program to set baseline concentrations.

**Distilled Water:** Commercially available water that has been distilled. Each batch of distilled water should be analyzed to set baseline concentrations.

**Hand Auger:** A sampling tool consisting of a metal tube with two sharpened spiral wings at the tip.

**Split-Spoon Sampler:** A sampling tool consisting of a thick-walled steel tube with a removable head and drive shoe. The steel tube splits open lengthwise when the head and drive shoe are removed.

**Scoop:** A sampling hand tool consisting of a small shovel- or trowel-shaped blade.

**Submersible Pump:** Groundwater sampling pump that consists of a rotor contained within a chamber and driven by an electric motor.

#### **4.0 RESPONSIBILITIES**

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Responsible for project implementation and coordination, selects project-specific drilling and sampling methods, and associated decontamination procedures with input from other key project staff, and appropriate oversight agencies.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer:** Implements the field program and supervises other sampling personnel, and ensures that SOPs are properly followed. Prepares daily logs of field activities.



**Field Sampling Technician (or other designated personnel):** Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks and is responsible for the decontamination of sampling equipment.

## **5.0 DECONTAMINATION PROCEDURES**

Drilling and sampling procedures require that decontaminated tools be employed in order to prevent cross-contamination. The decontamination procedures described below will be followed to ensure that only uncontaminated materials will be introduced to the subsurface during drilling and sampling. The equipment decontamination process will be undertaken before and after each use of the equipment and include either steam cleaning or washing. Steam cleaning of equipment, if used, will be performed at a temporary decontamination site. The flooring of the temporary decontamination site will be impermeable to water and large enough to contain the equipment and the rinsate produced.

If the quantity of water in the pad area exceeds its holding capacity, the water will be drummed temporarily until analytical results are obtained and the water can be properly disposed of. Steam cleaning will not be performed over bare ground, but will always be conducted so that rinsate can be collected and disposed of properly. Wherever applicable, equipment will be disassembled to permit adequate cleaning of the internal portions.

### **5.1 Drilling and Large Equipment**

The following procedure will be used for decontamination of large pieces of equipment. These include well casings, auger flights, drill pipes and rods, and those portions of the drill rig that may stand directly over a boring or well location, or that may come into contact with casing, auger flights, pipes, or rods.

- Establish a decontamination area large enough to contain the equipment and any decontamination waste
- Place equipment on sawhorse or equivalent, if possible.

- Steam clean the external surfaces and internal surfaces, as applicable, on equipment using high-pressure steam cleaner from an approved water source. If necessary, scrub using brushes and a phosphate-free detergent (e.g., Alconox<sup>TM</sup>), or equivalent laboratory-grade detergent until all visible dirt, grime, grease, oil, loose paint, rust, etc., have been removed.
- Rinse with potable water
- Remove equipment from decontamination pad and allow to air dry
- Record date and time of equipment decontamination

## **5.2 Soil and Groundwater Sampling Equipment**

The following procedure will be used to decontaminate sampling equipment such as split-spoon samplers; brass sleeves; continuous core barrels; scoops; hand augers; non-dedicated bailers; submersible pumps, bladder pumps; and other sampling equipment that may come into contact with samples. To minimize decontamination procedures in the field, dedicated equipment will be used wherever feasible:

- Wash and scrub equipment with phosphate-free, laboratory-grade detergent (e.g., Alconox<sup>TM</sup> or equivalent) and off-site distilled water
- Triple-rinse with distilled water
- Air dry
- Wrap in aluminum foil, or store in clean plastic bag or designated casing.

- Record date and time of equipment decontamination

Personnel involved in decontamination activities will wear appropriate protective clothing as defined in the project-specific health and safety plan.

### **5.3 Monitoring Equipment**

The following procedure will be used to decontaminate monitoring devices such as slug-test equipment, groundwater elevation and free product thickness measuring devices, and water quality checking instruments. Note that organic solvents can not be used to decontaminate free product measuring devices because they will cause damage to the probes. Spray bottles may be used to store and dispense distilled water.

- Wash equipment with laboratory-grade, phosphate-free detergent (e.g., Alconox™ or equivalent) and distilled water
- Triple-rinse with distilled water
- Store in clean plastic bag or storage case.
- Record date and time of equipment decontamination

## **6.0 QUALITY ASSURANCE AND QUALITY CONTROL**

All equipment decontamination must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein.

To assess the adequacy of decontamination procedures, field rinsate blanks may be collected. The specific number of rinsate blanks will be defined in a FSP or work plan or by the Project

Manager. In general, at least one field rinsate blank should be collected per sampling event or per day.

Rinsate blanks with elevated or detected contaminants will be evaluated by the Project Manager, who will relay the results to the site workers. Such results may be indicative of inadequate decontamination procedures that require corrective actions (e.g., retaining).

## **7.0 PROCEDURE FOR WASTE DISPOSAL**

All decontamination water that has come into contact with contaminated equipment will be handled, labeled, stored and disposed according to SOP 12. Unless otherwise specified in the FSP, waste generated from other sources and classified as non-hazardous waste (e.g., PPE, plastic sheeting, rope and misc. debris) will be disposed into trash receptacles.

## **8.0 REFERENCES**

Agency for Toxic Substances and Disease Registry. 2006. *Asbestos Exposure and Your Health*.

U.S. Environmental Protection Agency, *RCRA Ground-Water Monitoring: Draft Technical Guidance*, November 1992. Page 7-17.

Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007

OU3 SOP 8 (Rev. 0)

Title: SAMPLE HANDLING AND SHIPPING

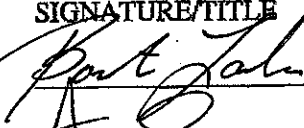
APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

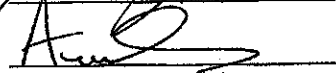
DATE

EPA Remedial Project Manager



9/26/07

SOP Author



9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--

## 1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-09, Sample Handling and Shipping, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. This SOP describes the requirements for sample handling, storage and shipping. The purpose of this SOP is to define sample management activities as performed from the time of sample collection to the time they are received by the laboratory.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## 3.0 DEFINITIONS

**Chain-of-Custody:** An accurate written record of the possession of each sample from the time of collection in the field to the time the sample is received by the designated analytical laboratory.

**Sample:** Physical evidence collected for environmental measuring and monitoring. For the purposes of this SOP, sample is restricted to solid, aqueous, air, or waste matrices. This SOP does not cover samples collected for lithologic description nor does it include remote sensing imagery or photographs (refer to SOP-9 for field documentation procedures).

**Sampler:** The individual who collects environmental samples during fieldwork.

## 4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, and etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** The Project Manager is responsible for ensuring that the requirements for sample management are included in the appropriate project plans. The Project Manager is responsible for coordinating sample management efforts with input from other key project staff and applicable government agencies.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader and/or Field Hydrogeologist, Geologist or Engineer:** Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

**Field Technician:** Responsible for sample collection, documentation, packaging, and shipping. Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks.

## 5.0 PROCEDURES

### 5.1 Applicability

The information in this SOP may be used by direct reference or incorporated into project-specific plans. Deviations or modifications to procedures addressed herein must be brought to the attention of, and approved by, applicable government agencies.

## 5.2 Sample Management

**Sample Containers:** The sample containers to be used will be dependent on the sample matrix and analyses desired, and are specified in the project FSP. Only certified pre-cleaned sample containers will be used. Sample containers will be filled with adequate headspace (approximately 10 percent) for safe handling upon opening, except containers for volatile organic compound (VOC) analyses, which will be filled completely with no headspace. This no-headspace requirement applies to both soil and groundwater samples.

Once opened, the containers will be used immediately. If the container is used for any reason in the field (e.g., screening) and not sent to the laboratory for analysis, it will be discarded. Prior to discarding the contents of the used container and the container, disposal requirements will be evaluated. When storing before and after sampling, the containers will remain separate from solvents and other volatile organic materials. Sample containers with preservatives added by the laboratory will not be used if held for an extended period on the job site or exposed to extreme heat conditions. Containers will be kept in a cool, dry place. For preserved samples (except VOCs), the pH of the sample will be checked following collection of the sample. If the pH is not at the required level, additional preservative (provided by the laboratory) will be added to the sample container.

**Numbering and Labeling:** Refer to OU3 SOP-9.

**Custody Seals.** Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposites sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

**Chain-of-Custody:** COC procedures require a written record of the possession of individual samples from the time of collection through laboratory analyses. A sample is considered to be in custody if it is:



- In a person's possession
- In view after being in physical possession
- In a secured condition after having been in physical custody
- In a designated secure area, restricted to authorized personnel

The COC record will be used to document the samples taken and the analyses requested. Refer to SOP-9 Attachment 2 for the OU3-specific COC form. Information recorded by field personnel on the COC record will include the following:

- Sample identifier (Index ID)
- Date and time of collection
- Sample matrix
- Preservation
- Type of analyses requested
- Unique COC number
- Lab being shipped to
- Signature of individuals involved in custody transfer (including date and time of transfer)
- Airbill number (if appropriate)
- Any comments regarding individual samples (e.g., organic vapor meter readings, special instructions).

COC records will be placed in a waterproof plastic bag (e.g., Ziploc®), taped to the inside lid of the cooler or placed at the top of the cooler, and transported with the samples. Signed airbills will serve as evidence of custody transfer between the field sampler and courier, as well as between the courier and laboratory. If a carrier service is used to ship the samples (e.g., Federal Express, etc.), custody will remain with the courier until it is relinquished to the laboratory. Upon receiving the sample cooler, a laboratory representative should sign in the receiving box of the COC, thus establishing custody. The sampler will retain copies of the COC record and airbill.

**Sample Preservation/Storage:** The requirements for sample preservation are dependent on the desired analyses and the sample matrix, and are specified in the FSP.

### 5.3 Sample Shipping

The methods and procedures described in this SOP were developed from these sources:

- 49 CFR 173. Shippers – Shippers – General Requirements for Shipping. United States Code of Federal Regulations available online at <http://www.gpoaccess.gov/cfr/index.html>
- 49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations available online at <http://www.gpoaccess.gov/cfr/index.html>
- ASTM D 4220. Standard Practice for Preserving and Transporting Soil Samples. American Society for Testing and Materials available online at <http://www.astm.org/>
- ASTM D 4840. Standard Practice for Sampling Chain-of-Custody Procedures. American Society for Testing and Materials available online at <http://www.astm.org/>

Procedures for packaging and transporting samples to the laboratory are dependent on the chemical, physical, and hazard properties of the material. The procedures may also be based on an estimation of contaminant concentrations/properties in the samples to be shipped. Samples will be identified as environmental samples, excepted quantities samples, limited quantities samples, or standard hazardous materials. Environmental samples are defined as solid or liquid samples collected for chemical or geotechnical analysis. Excepted quantities involve the shipment of a few milliliters of either an acid or base preservative in an otherwise empty sample container. Limited quantities are restricted amounts of hazardous materials that may be shipped in generic, sturdy containers. Standard hazardous material shipments require the use of stamped/certified containers. All samples will be packaged and shipped or hand delivered to the laboratories the same day of sample collection, unless otherwise specified in the project-specific FSPs.

The following paragraphs describe standard shipping procedures for different types of samples. Any exceptions to these procedures will be defined in the FSP. It is the responsibility of the sampler to refer to the U.S. Department of Transportation (DOT) (<http://hazmat.dot.gov/regs/rules.htm>) regulations when dealing with a substance not addressed in this SOP for requirements and limitations associated with the shipment.

### **Sample Shipping via Commercial Carrier:**

**Aqueous or Solid Samples:** Samples will be packaged and shipped to the laboratories the same day of sample collection, unless otherwise specified in the FSP and depending on holding time requirements for individual samples. For aqueous or solid samples that are shipped to the laboratory via a commercial carrier the following procedures apply:

- Sample labels will be completed and attached to sample containers.
- The samples will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler.
- For shipments containing samples for volatile organic analysis, include a trip blank.
- Ice in double Ziploc<sup>®</sup> bags (to prevent leakage) will be placed around, among, and on top of the sample bottles. Enough ice will be used so that the samples will be chilled and maintained at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  during transport to the laboratory. Dry ice or blue ice will not be used.
- To prevent the sample containers from shifting inside the cooler, the remaining space in the cooler will be filled with inert cushioning material, such as shipping peanuts, additional bubble pack, or cardboard dividers, such that the sample containers remain upright and do not break.
- Tape shut the cooler's drain plug
- The original copy of the completed COC form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid or placed at the top of the cooler.
- The lid will be secured by wrapping strapping tape completely around the cooler in two locations.

- Mark the cooler with arrow labels indicating the proper upright position of the cooler.
- Custody seals consisting of security tape with the date and initials of the sampler will be used on each shipping container to ensure custody. Two signed custody seals will be placed on the cooler, one on the front and one on the back.
- A copy of the COC record and the signed air bill will be retained for the project files.
- Affix a label containing the name and address of the shipper to the outside of the cooler

**Hand-Delivered Samples:** For aqueous or solid samples that will be hand carried to the laboratory, the same procedures apply.

**Excepted Quantities:** Usually, corrosive preservatives (e.g., hydrochloric acid, sulfuric acid, nitric acid, or sodium hydroxide) are added to otherwise empty sample bottles by the analytical laboratory prior to shipment to field sites. However, if there is an occasion whereby personnel are required to ship bottles with these undiluted acids or bases, the containers will be shipped in the following manner:

1. Each individual sample container will have not more than 30 milliliters of preservative.
2. Collectively, the preservative in these individual containers will not exceed a volume of 500 milliliters in the same outer box or package.
3. Despite the small quantities, only chemically compatible material may be placed in the same outer box, (e.g., sodium hydroxide, a base, must be packaged separately from the acids).
4. Federal Express will transport nitric acid only in concentrations of 40 percent or less.

5. A "Dangerous Goods in Excepted Quantities" label will be affixed to the outside of the outer box or container. Information required on the label includes:

- Signature of Shipper
- Title of Shipper
- Date
- Name and Address of Shipper
- Check of Applicable Hazard Class
- Listing of UN Numbers for Materials in Hazard Classes

**Limited Quantities:** Occasionally, it may become necessary to ship known hazardous materials, such as pure or floating product. DOT regulations permit the shipment of many hazardous materials in "sturdy" packages, such as an ice chest or cardboard box (not a specially constructed and certified container), provided the following conditions are met:

1. Each sample bottle is placed in a plastic bag, and the bag is sealed. Each VOC vial will be placed in a sealable bag. As much air as possible is squeezed from the bag before sealing. Bags may be sealed with evidence tape for additional security.
2. Or each bottle is placed in a separate paint can, the paint can is filled with vermiculite, and the lid is affixed to the can. The lid must be sealed with metal clips, filament, or evidence tape. If clips are used, the manufacturer typically recommends six clips.
3. The cans are placed upright in a cooler that has had the drain plug taped shut inside and outside, and the cooler is lined with a large plastic bag. Approximately 1 inch of adsorbent material sufficient to retain any liquid that may be spilled, is placed in the bottom of the liner. Only containers having chemically compatible material may be packaged in each cooler or other outer container.
4. The COC record is sealed inside a plastic bag and placed inside the cooler. The sampler retains one copy of the COC record. The laboratory will be notified if the

sample is suspected of containing any substance for which the laboratory personnel should take safety precautions.

5. The cooler is shut and sealed with strapping tape (filament type) around both ends. Two signed custody seals will be placed on the cooler, one on the front and one on the back. Additional seals may be used if the sampler and/or shipper consider more seals to be necessary. Wide, clear tape will be placed over the seals to ensure against accidental breakage.
6. The following markings are placed on the side of the cooler:
  - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA Dangerous Goods Regulations [DGR])
  - UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
  - Shipper's name and address
  - Consignee's name and address
  - The words "LIMITED QUANTITY"
  - Hazard Labels (Column E, List of Dangerous Goods, Section 4, IATA DGR)
  - Two Orientation (Arrow) labels placed on opposite sides.
7. The Airbill/Declaration of Dangerous Goods form is completed as follows:
  - Shipper's name and address
  - Consignee's name and address
  - Services, Delivery & Special Handling Instructions
  - Cross out "Cargo Aircraft Only" in the Transport Details Box
  - Cross out "Radioactive" under Shipment Type
  - Nature and Quantity of Dangerous Goods

- Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA DGR)
- Class or Division (Column C, List of Dangerous Goods, Section 4, IATA DGR)
- UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
- Packing Group (Column F, List of Dangerous Goods, Section 4, IATA DGR)
- Subsidiary Risk, if any (Column D, List of Dangerous Goods, Section 4, IATA DGR)
- Quantity and type of packing (number and type of containers: for example, "3 plastic boxes", and the quantity per container, "2 L", is noted as "3 Plastic boxes X 2 L" This refers to 3 plastic boxes (coolers are referred to as plastic boxes) with 2 liters in each box.
- Packing Instructions (Column G, List of Dangerous Goods, Section 4, IATA DGR).
- Note: Only those Packing Instructions in Column G that begin with the letter "Y" may be used. These refer specifically to the Limited Quantity provisions.
- Authorization (Write in the words Limited Quantity)
- Emergency Telephone Number (List 800-535-5053. This is the number for INFOTRAC.)
- Printed Name and Title, Place and Date, Signature.

**Standard Hazardous Materials:** Shipment of standard hazardous materials presents the most difficulty and expense. However, there may be occasion whereby a hazardous material cannot be shipped under the Limited Quantity provisions, (e.g., where there is no Packing Instruction in Column G, List of Dangerous Goods, IATA Dangerous Goods Regulations, that is preceded by the letter "Y").

In such cases, the general instructions noted above but for non-Limited Quantity materials will apply, with one important difference: standard hazardous materials shipment requires the use of certified outer shipping containers. These containers have undergone rigid testing and are, therefore, designated by a "UN" stamp on the outside, usually along the bottom of a container's side. The UN stamp is also accompanied by codes specifying container type, packing group rating, gross mass, density, test pressure, year of manufacturer, state of manufacturer, and manufacturer code name. The transport of lithium batteries in Hermit Data Loggers is an example of a standard hazardous material where only a designated outer shipping container may be used.

#### **5.4 Holding Times**

The holding times for samples will depend on the analysis and the sample matrix. Refer to the FSP for holding times requirements.

### **6.0 QUALITY ASSURANCE AND QUALITY CONTROL**

All sample shipments must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that handling and shipment documentation has been completed per this procedure and other procedures referenced herein.

### **7.0 DECONTAMINATION**

All shipment coolers shall be maintained clean of sampled material to avoid exposure during shipment. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

### **8.0 REFERENCES**

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.



Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, Draft, Appendix D, April 1980.

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Date: March 2, 2011

OU3 SOP 9 (Rev. 6)

Title: FIELD DOCUMENTATION

**APPROVALS:**

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	_____	_____
SOP Author	_____	_____

Revision Number	Date	Reason for Revision
0	09/26/2007	--
1	10/5/2007	<ul style="list-style-type: none"> <li>Add section for "Corrections and Modifications" and Field Modification Approval form (Attachment 3)</li> <li>Update Labeling section and COC (Attachment 2) to reflect non-asbestos analysis and container details</li> <li>Update FSDS forms (Attachment 1) based on field team input</li> </ul>
2	02/22/2008	<ul style="list-style-type: none"> <li>Incorporate changes to FSDS forms (Attachment 1) based on field input</li> <li>Remove OU3 phase specificity in SOP text</li> </ul>
3	05/29/2008	<ul style="list-style-type: none"> <li>Incorporate changes to FSDS forms (Attachment 1) based on field input</li> </ul>
4	06/30/2008	<ul style="list-style-type: none"> <li>Update Attachment 1 with all OU3 FSDS forms (including those used in Phase I and Phase II)</li> <li>Remove OU3 phase specificity in Attachments</li> </ul>
5	05/20/2009	<ul style="list-style-type: none"> <li>Add FSDS form for ABS Personal Air</li> <li>Add FSDS form for Small Mammal Tissue</li> <li>Modified COC to change medium code to "A-Air" to accommodate both ambient and activity-based sampling (ABS) air samples</li> <li>Added new media code for small mammal tissue "MT"</li> </ul>
6	02/25/2011	<ul style="list-style-type: none"> <li>Modify FSDS for surface water and sediment to include field for volume applied to syringe filter</li> </ul>

## 1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the required documentation to be completed by field personnel during field investigations. This SOP is based on MWH SOP-04, Field Documentation, Revision 1.0, March 2006, modified for use at the Libby Mine Site. Documentation in the form of field logbooks, reports, and forms shall be completed for every activity in the field. Records shall be maintained on a daily basis as the work progresses. All field documentation shall be accurate and legible because it is deliverable to the client as potentially a legal document.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the site health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006). All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan.

## 3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Selects project-specific field documentation with input from other key project staff.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer:** Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

**Field Technician (or other designated personnel):** Assists the FTL and/or field geologist, hydrogeologist, or engineer in the implementation of field tasks and field documentation.

**Field Sample/Data Manager:** Responsible for proper handling and shipping of all samples collected by the field crew, electronic data entry of field sample data sheet (FSDS) and chain-of-custody (COC) forms, and scanning/posting of field documentation PDFs (FSDS, COC, field logbooks, digital photographs) to a dedicated FTP site.

## **4.0 FIELD DOCUMENTATION PROCEDURES**

Field documentation serves as the primary foundation for all field data collected that will be used to evaluate the project site. There are two main forms of field documentation – field logbooks and FSDS forms. All field documentation shall be accurate, legible and written in indelible black or blue ink. Absolutely no pencils or erasures shall be used. Incorrect entries in the FSDS forms or field logbooks will be corrected by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction.

### **4.1 Field Logbooks**

The field logbook shall be a bound, weatherproof book with numbered pages, and shall serve primarily as a daily log of the activities carried out during the fieldwork. All entries shall be made in indelible black or blue ink. A field logbook shall be completed for each operation undertaken during the field tasks. To further assist in the organization of the field log books, the project name and the date shall be recorded on top of each page along with the significant

activity description (e.g., surface sample or soil boring number). All original field documentation shall be retained in the project files.

Skipped pages or blank sections at the end of a field log book page shall be crossed out with an "X" covering the entire page or blank section; "No Further Entries," initials, and date shall be written by the person crossing out the blank section or page. The responsible field team member shall write his/her signature, date, and time after the day's last entry.

Field activities vary from project to project; however, the concept and general information that shall be recorded are similar. The descriptions of field data documentation given below serve as an outline; individual activities may vary in documentation requirements. A detailed description of two basic example logbooks, suitable for documentation of field activities, is given below. These field logbooks include the FTL logbook and the field geologist/sampling team logbook.

**FTL Logbook:** The FTL's responsibilities include the general supervision, support, assistance, and coordination of the various field activities. As a result, a large portion of the FTL's day is spent rotating between operations in a supervisory mode. Records of the FTL's activities, as well as a summary of the field team(s) activities, shall be maintained in a logbook. The FTL's logbook shall be used to fill out daily/weekly reports and daily quality control reports (DQCRs), and therefore, shall contain all required information. Entries shall be preceded with time in military units for each observation. Items to be documented include:

- Record of tailgate meetings
- Personnel and subcontractors on job site and time spent on the site
- Field operations and personnel assigned to these activities
- Site visitors
- Log of FTL's activities: time spent supervising each operation and summary of daily operations as provided by field team members
- Problems encountered and related corrective actions
- Deviations from the sampling plan and reasons for the deviations
- Records of communications; discussions of job-related activities with the client, subcontractor, field team members, and project manager

- Information on addresses and contacts
- Record of invoices signed and other billing information
- Field observations

**Field Geologist/Sampling Team Logbook:** The field geologist or sampling team leader shall be responsible for recording the following information in a logbook:

- Health and Safety Activities
  - Calibration records for health and safety equipment (e.g., type of PID, calibration gas used, associated readings, noise dosimeters, etc.)
  - Personnel contamination prevention and decontamination procedures
  - Record of daily tailgate safety meetings
- Weather
- Calibration of field equipment
- Equipment decontamination procedures
- Personnel and subcontractors on job site and time spent on the site
- Station identifier
- Sampling activities
  - Sample location (sketch)
  - Equipment used
  - Names of samplers
  - Date and time of sample collection
  - Sample interval
  - Number of samples collected
  - Analyses to be performed on collected samples
- Disposal of contaminated wastes (e.g., PPE, paper towels, Visqueen, etc.)
- Field observations
- Problems encountered and corrective action taken
- Deviations from the sampling plan and reason for the deviations
- Site visitors

## 4.2 Field Sample Documentation

**Sample Labels:** A unique sample identification label shall be affixed to all sample containers. All samples will be labeled in a clear, precise way for proper identification in the field and for tracking in the laboratory. At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number, referred to as the Index ID. The Index ID for all samples collected as part of OU3 sampling activities will have a two-character prefix specific to the sampling Phase (e.g., Phase 1 samples will have a “P1” prefix, P1-12345) as specified in the applicable SAP. Index ID labels will be ½ inch x 1 ¾ inch in size and pre-printed for use in the field. For each Index ID, multiple labels will be printed to allow for multiple containers of the same sample (i.e., for different analyses).

Index ID Label Example:

**P1-12345**

Each collection container will be labeled with a container label that enables the field team member to record the container-specific details, such as the method of sample preparation (e.g., filtered/unfiltered), method of preservation, and the analytical methods that will be requested. Container labels will be 2 inch x 4 inch in size and pre-printed for use in the field. Any container-specific information shall be written in indelible ink.

Container Label Example:

Index ID:		Date/Time: _____	
Media (circle one):    AQ    SO    A    BK    DB    TC    MT			
For AQ, Filtered? (circle one):    Yes    No			
Container: _____			
Preservation: _____			
Analyses: _____			
_____			

Media acronyms: AQ – aqueous media, SO – solid media, A – air,  
BK – tree bark, DB – organic debris, TC – tree age core, MT – mammal tissue



After labels have been affixed to the sample container, the labels will be covered with clear packaging tape to ensure permanence during shipping.

Any unused Index ID labels should be crossed out to avoid the possibility of using unused labels for a different sample.

**Field Sample Data Sheet (FSDS) Forms:** Data regarding each sample collected as part of the OU3 sampling will be documented using Libby-specific FSDS forms (provided as Attachment 1). These FSDS forms are medium-specific and designed to facilitate data entry of station location, sample details, and field measurements needed for the OU3 investigation.

In the field, one field team member will be responsible for recording all sample details onto the appropriate FSDS form. At the time of sample labeling, one Index ID label will be affixed to the FSDS form in the appropriate field. All written entries on the FSDS form shall be accurate, legible and written in indelible black or blue ink.

Once the FSDS form is complete, written entries will be checked by a second field team member. These two field team members will initial the bottom of the FSDS form in the appropriate field to document who performed the written data entry and who performed the QC check of the FSDS form.

On a weekly basis (or more frequently as conditions permit), information from the hard copy FSDS form will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate. The Field Sample/Data Manager and the FTL will initial in the appropriate field on the paper FSDS form to document who performed the data entry into the database and who performed the QC check.

### **4.3 Photologs**

Photologs are often used in the field to document site conditions and sample location characteristics. While photographs may not always be required, they shall be used wherever

applicable to show existing site conditions at a particular time and stage of the investigation or related site activity. Photolog information shall include:

- station location identifier
- Index ID (if applicable)
- date and time of photo
- direction/orientation of the photo
- description of what the photo is intended to show

An engineer's scale or tape shall be included in any photographs where scale is necessary. Any wasted frames or images in a roll of film or sequence of digital images shall be so noted in the field logbook.

#### **4.4 Chain-of-Custody Records**

**Custody Seals:** Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposites sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

**Chain-of-Custody Forms:** COC procedures allow for the tracking of possession and handling of individual samples from the time of field collection through to laboratory analysis. Documentation of custody is accomplished through a COC form that lists each sample and the individuals responsible for sample collection and shipment, sample preparation, and receipt by the analytical laboratory. The COC form also documents the analyses requested for each sample. Whenever a change of custody takes place, both parties will sign and date the COC form, with the relinquishing party retaining a copy of the form. The party that accepts custody will inspect the COC form and all accompanying documentation to ensure that the information is complete and accurate. Any discrepancies will be noted on the COC form. Shipping receipts shall be signed and filed as evidence of custody transfer between field sampler(s), courier, and laboratory.

Attachment 2 provides an example of the COC form that will be used for all samples collected as part of OU3 sampling. This form will be printed as a carbonless triplicate form to facilitate retention of COC copies by relinquishing parties. As seen, the COC form includes the following information:

- sample identifier (Index ID)
- date and time of collection
- method of sample preparation and preservation
- number of sample containers
- analyses requested
- shipping arrangements and airbill number, as applicable
- recipient laboratories
- signatures of parties relinquishing and receiving the sample

On a daily basis, the Field Sample/Data Manager will package samples for shipping, complete hard copy COC forms, and ship all samples as outlined in SOP No. 8. On a daily basis, information from the hard copy COC form necessary for sample tracking will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate.

## **5.0 FIELD DATA TRANSMITTAL**

Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site daily. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS\_20090831.pdf).

Electronic copies of all digital photographs will also be posted weekly (or more frequently as conditions permit) to the project-specific FTP site. File names for digital photographs will include the station identifier, the sample date, and photograph identifier (e.g., ST-1\_20090831\_12459.tif).

A copy of the field-specific OU3 database will be posted to the project-specific FTP site on a weekly basis (or more frequently as conditions permit). The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB\_20090831.mdb).

## **6.0 CORRECTIONS AND MODIFICATIONS**

### **6.1 Field Deviations and Modifications**

It is recognized that deviations and modifications from the standard operating procedures may be necessary based on site conditions. Any requested field modifications will be submitted by Robert Marriam (Remedium Group, Inc. - W.R. Grace contractor) to Bonita Lavelle (EPA Region 8 - Remedial Project Manager) for review and approval. All modification requests will be recorded in a Field Modification Approval Form (see Attachment 3).

### **6.2 Corrections to Hard Copy Forms**

If an error is identified on an FSDS or COC form prior to entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. Data entry into the field-specific OU3 database and scanning/posting of the hard copy forms should proceed following the data entry procedures described above.

If an error is identified on an FSDS or COC form after entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. The corrected form should be scanned and posted to the project-specific FTP site. File names for corrected FSDS forms will include the Index ID of the corrected sample to facilitate document organization (e.g., FSDS\_C\_P1-12345.pdf). File names for corrected COC

forms will include the COC ID of the corrected COC form to facilitate document organization (e.g., COC\_C\_OU3-36512.pdf). Necessary data corrections will be made to the master OU3 database by the database manager.

If changes are made to a COC form, the analytical laboratory should be provided with a corrected COC form.

## **7.0 REFERENCES**

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992.

**ATTACHMENT 1**

**OU3 FIELD SAMPLE DATA SHEET (FSDS) FORMS**

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2

## STATIONARY AMBIENT AIR MONITOR

Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_

☐ Check box if GPS information  
has been recorded previously

Station ID: \_\_\_\_\_ Station Comments: \_\_\_\_\_

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

X coord: \_\_\_\_\_ Y coord: \_\_\_\_\_ Elevation: \_\_\_\_\_ m

Sampling Team: \_\_\_\_\_ Sampler Initials: \_\_\_\_\_

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID	AFFIX LABEL HERE	AFFIX LABEL HERE	AFFIX LABEL HERE
Sample Height (ft)			
Location Description			
Field QC Type (circle)	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID:	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID:	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID:
Matrix Type	Outdoor	Outdoor	Outdoor
Flow Meter Type	Rotameter	Rotameter	Rotameter
Archive blank (circle)	Yes No	Yes No	Yes No
Pump ID Number			
Flow Meter ID Number			
Start Date (mm/dd/yy)			
Start Time (hh:mm)			
Start Counter			
Daily Flow Check:	Check1 Time Flow	Check1 Time Flow	Check1 Time Flow
Record time (hh:mm) and flow rate (L/min) in fields provided	Check2	Check2	Check2
	Check3	Check3	Check3
	Check4	Check4	Check4
Stop Date (mm/dd/yy)			
Stop Time (hh:mm)			
Stop Counter			
Pump fault? (circle)	Yes No	Yes No	Yes No
Stop Flow (L/min)			
Field Comments			
Cassette Lot Number: _____			
Entered By (Provide initials):	Validated By (Provide initials):		

For Data Entry Completion (Provide Initials)

Completed by:

QC by:

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2

## FOREST SOIL AND TREE BARK

Field Logbook No: \_\_\_\_\_

Page No: \_\_\_\_\_

Station ID: \_\_\_\_\_

Sampling Date: \_\_\_\_\_

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

X coord: \_\_\_\_\_ Y coord: \_\_\_\_\_ Elevation: \_\_\_\_\_m

Sampling Team: \_\_\_\_\_ Sampler Initials: \_\_\_\_\_

Station Comments: \_\_\_\_\_

**TREE BARK SAMPLES**

Index ID: _____	<b>Field QC Type (circle one):</b> FS (field sample) FD (field duplicate) For FD, Parent ID: _____	<b>Sample Area (cm<sup>2</sup>):</b> _____	<b>Tree Species:</b> _____  <b>Collection Height (ft):</b> _____	<b>Age Core Collected? (circle one):</b> Y    N
Index ID: _____	<b>Field QC Type (circle one):</b> FS (field sample) FD (field duplicate) For FD, Parent ID: _____	<b>Sample Area (cm<sup>2</sup>):</b> _____	<b>Diameter* (in):</b> _____	
Field Comments:				
Entered by (Provide initials):			Validated by (Provide initials):	

\*Measured with "D-tape"

**FOREST SOIL SAMPLES**

Index ID: _____	<b>Field QC Type (circle one):</b> FS (field sample) FD (field duplicate) For FD, Parent ID: _____	<b>Bulk Soil Description</b>		<b>Organic Debris Collected? (circle one):</b> Y    N
		<b>Depth (in)</b> <b>Start:</b> _____ <b>End:</b> _____	<b>Sample Type:</b> Grab Composite <b># of Comp.:</b> ____	
Index ID: _____	<b>Field QC Type (circle one):</b> FS (field sample) FD (field duplicate) For FD, Parent ID: _____	<b>Bulk Soil Description</b>		<b>Organic Debris Collected? (circle one):</b> Y    N
		<b>Depth (in)</b> <b>Start:</b> _____ <b>End:</b> _____	<b>Sample Type:</b> Grab Composite <b># of Comp.:</b> ____	
Index ID: _____	<b>Field QC Type (circle one):</b> FS (field sample) FD (field duplicate) For FD, Parent ID: _____	<b>Bulk Soil Description</b>		<b>Organic Debris Collected? (circle one):</b> Y    N
		<b>Depth (in)</b> <b>Start:</b> _____ <b>End:</b> _____	<b>Sample Type:</b> Grab Composite <b># of Comp.:</b> ____	
Field Comments:				
Entered by (Provide initials):			Validated by (Provide initials):	

For Data Entry Completion (Provide Initials)

Completed by

QC by



☐ Check box if GPS information has been recorded previously

Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_

Station ID: \_\_\_\_\_ Station Alias: \_\_\_\_\_ Sampling Date: \_\_\_\_\_

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

X coord:                      Y coord:                      Elevation (meters):

Sampling Team: \_\_\_\_\_ Sampler Initials: \_\_\_\_\_

Station Comments:

<b>Well:</b> Measuring Point (MP) of Well: _____ MP Units: _____ Screened Interval (ft BGL): _____ Filter Pack Interval (ft BGL): _____ Casing Stickup (ft) _____ Sample Intake Depth (ft BMP): _____	<b>Calibration:</b> <input type="checkbox"/> Daily Verification    Weekly Date: _____ VOA Vial pH: _____ Other pH: _____
---	--

**Purge Method:**

☐ Dedicated Submersible (SP)

☐ Portable Bladder (BP)

☐ Peristaltic (PP)

☐ Portable Submersible (SP)

☐ Dedicated Bailer (B)

☐ Grab (G)

☐ Dedicated Bladder (SP)

☐ Disposable Bailer (B)

☐ Other: \_\_\_\_\_

**Purge:**

Starting Water Level (ft BMP): \_\_\_\_\_ Total Depth (ft BGL): \_\_\_\_\_ Water Column Height (ft): \_\_\_\_\_

Casing Diameter (in ID): \_\_\_\_\_ Multiplication Factor: \_\_\_\_\_ Casing Volume (gal): \_\_\_\_\_ 2X: \_\_\_\_\_ 3X: \_\_\_\_\_ 4X: \_\_\_\_\_

Water Level (ft BMP) at End of Purge: \_\_\_\_\_ Total Depth (ft BMP) at End of Purge: \_\_\_\_\_

**Multiplication Factors:**

1" = 0.04    2" = 0.16

3" = 0.37    4" = 0.65

6" = 1.47

[illegible]

Data Item	Sample 1	Sample 2	Sample 3
Index ID	AFFIX LABEL HERE	AFFIX LABEL HERE	AFFIX LABEL HERE
Field QC Type (circle one):	FS   SP   FD   MS   MSD PE   EB   FB   TB Parent ID: _____	FS   SP   FD   MS   MSD PE   EB   FB   TB Parent ID: _____	FS   SP   FD   MS   MSD PE   EB   FB   TB Parent ID: _____
Field Comments:			
Cooler:			
Entered by (Provide initials):		Validated by (Provide initials):	

<b>Note:</b>	<b>FS</b>	Field Sample	<b>SP</b>	Field Split Sample	<b>FD</b>	Field Duplicate Sample
	<b>TB</b>	Trip Blank Sample	<b>MS</b>	Matrix Spike Sample	<b>MSD</b>	Matrix Spike Duplicate Sample
	<b>FB</b>	Field Blank Sample	<b>EB</b>	Equipment Decon Blank Sample	<b>PE</b>	Performance Evaluation Sample

For Data Entry Completion (Provide Initials)	Completed by	QC by
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## LIBBY OU3 FIELD SAMPLE DATA SHEET

### ACTIVITY-BASED SAMPLING (ABS) PERSONAL AIR MONITOR

ABS Area: <b>ABS-</b> _____		Sampling Date: _____		Sampling Team: <u>MWH</u>	
Person #1 Name: _____		Index ID: _____		AFFIX LABEL HERE	
Person #2 Name: _____		Index ID: _____		AFFIX LABEL HERE	
AFFIX LABEL HERE					
Field Blank Index ID: _____		Cassette Lot Number: _____			
Field Logbook Number: _____		Field Logbook Pages: _____			

ABS Activity	Activity Sample Time (hh:mm)		Rotometer Flow (L/min)			
			Person #1		Person #2	
	Start	Stop	Start	Stop	Start	Stop
ATV Riding						
Hiking						
Sawing/Stacking						
Raking/Digging						
Fire						

Person #1    Pump ID No.: \_\_\_\_\_    Rotometer ID No.: \_\_\_\_\_    GPS ID No.: \_\_\_\_\_  
 Person #2    Pump ID No.: \_\_\_\_\_    Rotometer ID No.: \_\_\_\_\_    GPS ID No.: \_\_\_\_\_

**Field Comments:**

*Weather Description--*

*Other--*

Field Data Entered by: _____	Field Entries Checked by: _____
------------------------------	---------------------------------

Database Entry: _____	Database QC: _____
-----------------------	--------------------

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2

## SOIL-LIKE MATERIALS

Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_

Station ID: \_\_\_\_\_ Sampling Date: \_\_\_\_\_

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

Sampling Team: \_\_\_\_\_ Sampler Initials: \_\_\_\_\_

Station Comments: \_\_\_\_\_

Data Item	Sample 1	Sample 2	Sample 3
<b>Index ID</b>	<i>AFFIX LABEL HERE</i>	<i>AFFIX LABEL HERE</i>	<i>AFFIX LABEL HERE</i>
Matrix (circle one):	Surface Soil      Tailings Waste Rock      Roadway Other _____	Surface Soil      Tailings Waste Rock      Roadway Other _____	Surface Soil      Tailings Waste Rock      Roadway Other _____
Sample Time (hh:mm)			
Sample Type (circle one):	Grab      Composite  # of Comp: _____	Grab      Composite  # of Comp: _____	Grab      Composite  # of Comp: _____
Sample Depth	Start Depth (in): _____  End Depth (in): _____	Start Depth (in): _____  End Depth (in): _____	Start Depth (in): _____  End Depth (in): _____
Field QC Type (circle one):	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____
Transect Start Location or Grab Sample Location	X coord: _____ m  Y coord: _____ m  Elevation: _____ m	X coord: _____ m  Y coord: _____ m  Elevation: _____ m	X coord: _____ m  Y coord: _____ m  Elevation: _____ m
Transect End Location	X coord: _____ m  Y coord: _____ m  Elevation: _____ m	X coord: _____ m  Y coord: _____ m  Elevation: _____ m	X coord: _____ m  Y coord: _____ m  Elevation: _____ m
Field Comments:			
Cooler:			
<b>Entered by (Provide initials):</b>		<b>Validated by (Provide initials):</b>	

For Data Entry Completion (Provide Initials)

Completed by

QC by

**Station ID:** \_\_\_\_\_ **Sampling Date:** \_\_\_\_\_  
**Field Logbook ID:** \_\_\_\_\_ **Logbook Page No:** \_\_\_\_\_  
**GPS Coordinate System:** UTM Zone 11 North, NAD83 datum, meters  
***For New Stations Only:*** **X coord:** \_\_\_\_\_ **Y coord:** \_\_\_\_\_ **Elev:** \_\_\_\_\_  
**Sampling Team:** **MWH** **Samplers Initials:** \_\_\_\_\_

Time Measured (hh:mm)	Temp. (°C)	pH	Specific Conductance (mS/cm Auto-comp @ 25°C)	Diss. O <sub>2</sub> (mg/L)	ORP (mV)	Turbidity (NTU)

WATER COLLECTION							
Index ID							
Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: Field Sample Media : <input type="checkbox"/> Sediment <input type="checkbox"/> SW (aqueous) <input type="checkbox"/> SW (filter) - Vol. Applied _____ mL	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____				
Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: Field Sample Media : <input type="checkbox"/> Sediment <input type="checkbox"/> SW (aqueous) <input type="checkbox"/> SW (filter) - Vol. Applied _____ mL	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____				
Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: Field Sample Media : <input type="checkbox"/> Sediment <input type="checkbox"/> SW (aqueous) <input type="checkbox"/> SW (filter) - Vol. Applied _____ mL	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____				
Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: SP FD MS MSD PE FB TB EB Media : <input type="checkbox"/> Sediment <input type="checkbox"/> SW (aqueous) <input type="checkbox"/> SW (filter) - Vol. Applied _____ mL	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____				

COMMENTS	

Field Data Entered by:		Field Entries Checked by:	
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Database QC:

## LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) SMALL MAMMAL TISSUE COLLECTION

Field Logbook ID: \_\_\_\_\_ Logbook Page No: \_\_\_\_\_

Necropsy Date: \_\_\_\_\_ Personnel Initials: \_\_\_\_\_

 Small Mammal Field ID: SM- \_\_\_\_\_ - \_\_\_\_\_ - \_\_\_\_\_ - \_\_\_\_\_ Animal Weight (grams): \_\_\_\_\_ (w/o fetuses if pregnant)  
[SM - station ID - transect ID - trap# - animal#]

General Necropsy Comments: \_\_\_\_\_

	TISSUE #1	TISSUE #2	TISSUE #3	TISSUE #4
Tissue Type (circle one):	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____
Weight (mg):				
Index ID:	<i>Affix Label Here</i>	<i>Affix Label Here</i>	<i>Affix Label Here</i>	<i>Affix Label Here</i>
Field QC Type (circle one):	FS    FD    TB	FS    FD    TB	FS    FD    TB	FS    FD    TB
Tissue Comments:				

	TISSUE #5	TISSUE #6	TISSUE #7	TISSUE #8
Tissue Type (circle one):	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____
Weight (mg):				
Index ID:	<i>Affix Label Here</i>	<i>Affix Label Here</i>	<i>Affix Label Here</i>	<i>Affix Label Here</i>
Field QC Type (circle one):	FS    FD    TB	FS    FD    TB	FS    FD    TB	FS    FD    TB
Tissue Comments:				

	TISSUE #9	TISSUE #10	TISSUE #11	TISSUE #12
Tissue Type (circle one):	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____	TY    AR    ES ST    SIN    LIN LU    EY    CAR Other: _____
Weight (mg):				
Index ID:	<i>Affix Label Here</i>	<i>Affix Label Here</i>	<i>Affix Label Here</i>	<i>Affix Label Here</i>
Field QC Type (circle one):	FS    FD    TB	FS    FD    TB	FS    FD    TB	FS    FD    TB
Tissue Comments:				

Tissue Type Descriptors: TY = thyroid; ARD = adrenal gland; ES = esophagus; SIN = small intestine; LIN = large intestine; LU = lung; EY = eyeball; CAR = carcass  
 Field QC Type Descriptors: FS = Field Sample; FD = Field Duplicate; TB = Tissue Blank

For Data Entry Completion (Provide Initials)

Completed by

QC by

**ATTACHMENT 2**

**OU3 CHAIN OF CUSTODY FORM**

## COC No. \_\_\_\_\_

PAGE: \_\_\_\_\_ OF: \_\_\_\_\_

METHOD OF SHIPMENT: \_\_\_\_\_ CARRIER/WAYBILL NO.: \_\_\_\_\_ DESTINATION: \_\_\_\_\_

[illegible]

			TOTAL NUMBER OF CONTAINERS	LABORATORY COMMENTS/CONDITION OF SAMPLES					Cooler Temp:
RELINQUISHED BY:			DATE	TIME	RECEIVED BY:				
SIGNATURE	PRINTED NAME	COMPANY			SIGNATURE	PRINTED NAME	COMPANY		

Notes --

(d) Preparation by ISSI-LIBBY-01 and analysis by SRC-LIBBY-01 (PLM-Grav) and SRC-LIBBY-03 (PLM-VE)

(e) *In accordance with procedures in Phipps (1985).*

**DISTRIBUTION:** PINK: Field Copy YELLOW: Laboratory Copy WHITE: Return to Originator

**ATTACHMENT 3**

**OU3 FIELD MODIFICATION APPROVAL FORM**



**FIELD MODIFICATION APPROVAL FORM**

**LFM-OU3-\_\_\_\_\_**

*Libby OU3 Phase \_\_\_\_ Sampling & Analysis Plan*

Requested by:\_\_\_\_\_ Date:\_\_\_\_\_

Description of Deviation:

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☐ EPA Region 8 has reviewed this field modification approves as proposed.

☐ EPA Region 8 has reviewed this field modification and approves with the following exceptions:

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☐ EPA Region 8 has reviewed this field modification and does not agree with the proposed approach for the following reasons:

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\_\_\_\_\_  
Christina Progross, EPA RPM

\_\_\_\_\_  
Date

LIBBY SUPERFUND SITE OPERABLE UNIT 3 STANDARD OPERATING PROCEDURE

Libby Site OU3 SOP No. 10

Rev. No. 1

Date: January 31, 2008

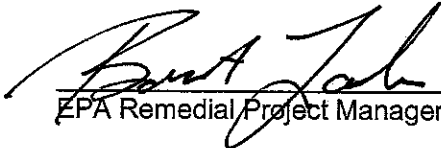
Page 1 of 37

Date: January 31, 2008

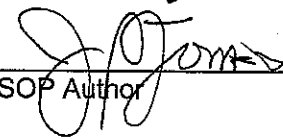
OU3 SOP 10 (Rev 1)

**Title: FIELD EQUIPMENT CALIBRATION**

**APPROVALS:**

  
\_\_\_\_\_  
EPA Remedial Project Manager

3/20/08  
\_\_\_\_\_  
Date

  
\_\_\_\_\_  
SOP Author

3/20/2008  
\_\_\_\_\_  
Date

Revision Number	Date	Reason for Revision
0	9/26/07	
1	1/31/08	Update based on field team input

**LIBBY ASBESTOS SUPERFUND SITE OU3**  
**STANDARD OPERATING PROCEDURE No.10**  
**FIELD EQUIPMENT CALIBRATION**

**1.0 INTRODUCTION**

This standard operating procedure (SOP) provides general guidelines on calibration and operating procedures for typical field equipment. This SOP is based on two documents: (1) MWH FMC SOP-01, Field Equipment Calibration, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3 and (2) USGS Survey Techniques of Water-Resources Investigations, Book 9. Field monitoring instruments are used to measure chemical parameters in situ and when data quality objectives specify screening-level analytical support. Screening-level data are collected for on-site, real-time measurements; evaluation of existing conditions; refinement of sampling locations; and health and safety evaluations. Field measurements are generally used to refine sampling programs and to estimate the extent of contamination at the site. This type of support also provides real-time data for health and safety purposes.

The purpose of this SOP is to define the calibration and operating procedures for equipment used for field monitoring.

**2.0 DEFINITIONS**

**Conductivity:** Is a measure of the quantity of electricity transferred across a unit area, per unit potential gradient, per unit time. Conductivity is measured by dipping a probe directly into the water source or into a separate sample aliquot.

**Dissolved Oxygen (DO):** Is a measure of the quantity of oxygen dissolved in water. DO data is collected in the field using direct measurement probes.

**pH:** Is measured directly using a probe and is the acidity or alkalinity of a solution; numerically equal to 7 for neutral solutions, increasing with increasing alkalinity and decreasing with increasing acidity.

**Temperature:** An indicator of the thermal energy contained in a solid or fluid. Units are degrees Centigrade (°C) or degrees Fahrenheit (°F). Temperature measurements are made with a mercury-filled thermometer, bimetallic-element thermometer, or electrical thermistor.

**Turbidity:** a measure of cloudiness in water due to suspended and colloidal organic and inorganic material. Turbidity is measured by using a field portable nephelometer capable of reading down to 0.1 Nephelometric Turbidity Units (NTU).

### 3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Responsible for identifying the appropriate equipment necessary for adequate site characterization and the requirements for the project-specific tasks.

**Quality Control Manager:** Performs field program audits and ensures project data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer:** Implements the field program, and supervises other field staff to ensure proper calibration and use of field equipment through the duration of the project.

**Field Technician (or other designated personnel):** Responsible for providing requested instrumentation and basic instructions on its calibration and use. Assists the FTL with the implementation of tasks and is responsible for regular equipment maintenance and calibration.

## **4.0 GUIDELINES**

This SOP provides a summary of the calibration and operating procedures in accordance with the various manufacturers' instruction manuals, which accompany each piece of equipment. This SOP will be reviewed and used in conjunction with the manufacturer's instruction manual by field team members when using field equipment.

### **4.1 APPLICABILITY**

Field equipment must be kept in designated cases, packaged properly, and secured during transport to prevent equipment damage, which may result in inaccurate readings. Decontaminate and calibrate all equipment prior to use. As part of the calibration process, standard laboratory procedures of decontamination shall be followed; prior to calibration and between calibration buffers, solution vessels and probes shall be rinsed a minimum of three times with distilled/deionized (DI) water and a minimum of one time with the calibration buffer solution or sample solution.

Always calibrate meters according to the manufacturer's instructions before the start of each workday and whenever equipment drift is suspected. Consult the specific instruments' instruction manual for further calibration details.

### **4.2 pH METERS**

Determining pH is critical for predicting and interpreting the reactions and migration of dissolved chemical constituents in groundwater or surface water. Whenever groundwater or surface water samples are collected, pH may be measured using a flow-

through type meter or another type of pH probe. Meters used will have temperature and slope adjustments and a repeatability of  $\pm 0.01$  standard pH units.

**4.2.1 Calibration:** Calibration standard buffers shall not be used beyond the date specified by the manufacturer, and calibration standard buffers shall be stored in a manner that protects the integrity and precision of the solution. Proper decontamination of equipment shall be performed following standard lab practices (refer to section 4.1) prior to calibration.

Two pH standard buffers will be used for calibration of the electrode and are to bracket the anticipated pH of the water samples. For example, if the anticipated pH of sample water is 6, calibration will be conducted with pH 4 and pH 7 buffer solutions; for an anticipated pH of 8, calibrate with pH 7 and pH 10 buffers. Three buffer solutions can be used to calibrate over a larger pH range. Because pH is temperature dependent, buffers and samples should be kept at similar temperature. The temperature of buffer solutions must be known, and temperature-correction factors must be applied before calibration adjustments are made. Theoretically, buffer solutions are stable indefinitely; however, they are susceptible to contamination. Therefore, old, partially full bottles will be replaced and solutions will not be used past the manufacturer's recommended expiration date. The instrument calibration will be checked periodically against a standard solution.

Meters with microprocessors have reliable autocalibration functions and will automatically compensate for buffer temperatures and indicate Nernst slope. For such meters, follow the manufacturer's calibration instructions precisely and completely.

- Check the records of electrode performance before each calibration and sampling event. Electrode response is optimum between approximately 98 percent and 99.5 percent. A slope of 94 percent indicates possible electrode deterioration. Do not use the electrode if the response slope is below 90 percent.
- Calibrate or check the temperature sensor calibration at least annually, and tag the sensor with the date of in-house certification. Do not use the automatic

temperature compensating function of a pH meter if it has not been certified within the past 12 months.

- Record calibration in the instrument log book and on field forms at the time of instrument calibration.

**Procedure:** Calibration and operating procedures differ with instrument systems—always check the manufacturer's instructions.

1. Equilibrate Equipment to Temperature (this is recommended, even if using an automatic compensating meter).

a. Bring the pH buffers, thermometer (if necessary), container, and electrode to the temperature of the sample.

- To equilibrate to stream temperature, place the buffer bottles in a minnow bucket or mesh bag and suspend them in the stream.

b. Allow 15 to 30 minutes for the buffers to adjust to the sample temperature.

- When making temperature corrections, use the correction factors provided by the buffer manufacturer (temperature coefficients can vary with buffer manufacturer).

2. Inspect the pH Electrode.

- a. Check for damage to the electrode bulb, body, or cables.
- b. Rinse any precipitate off of the electrode with DI water (the measurement can be affected if precipitate falls into the buffer or sample).
- c. Slide the protective sleeve up or down to uncover the filling hole.
- d. Gently shake or tap the electrode to dislodge and remove air bubbles trapped in the sensing tip of the electrode and to remove excess deionized water. Do not wipe the electrode.

3. Calibration Rinse.

- a. Rinse the electrode, thermometer or automatic temperature compensating (ATC) sensor, and a container large enough to hold the sensors and buffer with pH buffer 7. Discard the used pH buffer into a waste container.
- b. Pour fresh pH 7 buffer into the buffer-rinsed container that holds the electrode and thermometer. Allow the instruments to equilibrate for 1 minute (if necessary), then discard the buffer into a waste container.

#### 4. Calibration.

- a. Pour fresh pH 7 buffer into the container that holds the electrode and thermometer or ATC sensor.
  - The bulb of the pH electrode must not touch the bottom or side of the container.
  - Add enough pH buffer to cover the reference junction.
- b. Swirl the sample gently or stir carefully with the electrode. If using a magnetic stirrer, stir slowly enough so that a vortex is not created. Place a thin piece of insulating material (styrofoam or cardboard) between the magnetic stirrer and beaker to prevent transfer of heat to the buffer solution.
- c. Measure the temperature of the buffer solution; remove the thermometer (it is not necessary to remove the ATC sensor).
- d. Determine the theoretical pH of the buffer from the temperature-correction tables.
- e. Note and record the pH temperature readings. Adjust the meter reading to the pH value using the "standardize" function on the meter (usually a knob or pressure pad). Record the adjusted pH value for the 7.0 buffer and associated millivolt reading.
- f. Remove the electrode and ATC sensor (some instruments require that the meter be switched to the standby or off position before removing the electrode from the solution).



- Repeat the calibration steps using fresh portions of reference buffer solution until two successive readings are obtained at the adjusted pH value for pH 7 buffer without further adjustment to the system.
- Discard the used pH 7 buffer into a waste container.

#### 5. Slope Adjustment Rinse.

- a. Rinse the electrode and thermometer or ATC sensor thoroughly with DI water.
- b. Rinse a clean container, electrode, and thermometer with a second buffer (usually pH 4 or 10) that brackets the expected pH value of the sample; discard the used buffer into a waste container.
- c. Pour the second buffer into a container holding the electrode and thermometer or ATC sensor. Allow the temperature to equilibrate for 1 minute, then discard the used buffer into a waste container.

#### 6. Slope Adjustment. \*\*\*Note: For most modern meters, this step is automated—if so, skip to step seven.

- a. Pour a fresh portion of the second pH buffer into a container holding the electrode and thermometer or ATC sensor.
- b. Stir slowly (no vortex) or swirl manually. Follow the directions in 4b, above.
- c. Measure the temperature and pH of the buffer solution and check the pH value of the buffer on temperature coefficient tables. Record the pH and temperature readings.
- d. Adjust the slope to the value of the second pH buffer at known temperature. (Some meters have separate slope-adjustment knobs, pressure pads, or other devices, whereas others have to be adjusted by use of a temperature knob.) Record the adjusted pH value and associated millivolt reading.
- e. Discard the used buffer into a waste container.
- f. Repeat steps 6(a) through 6(e) using successive portions of the buffer solution until two successive readings are obtained without further adjustment.

7. Rinse the electrode and thermometer or ATC sensor thoroughly with DI water.
8. If using a non-compensating or non-automated meter, repeat the calibration rinse (step 3) and calibration procedures [steps 4(a) through 4(d)] to ensure that the slope adjustments did not affect the calibration adjustment.
  - This step is a check only; no adjustment should be needed, but the result should be recorded. If adjustment is needed, repeat the entire calibration procedure.
  - If adjustment is still needed, a systematic problem is likely (see 6.4.4). Inspect the instrument system, clean the electrode or add filling solution, or use a spare electrode or meter.
9. Calibration Check Rinse.
  - a. Rinse the electrode and thermometer or ATC sensor with DI water.
  - b. Rinse another clean container, electrode, and thermometer with a third buffer (pH 4 or 10) and discard the used buffer into a waste container.
  - c. Pour the third buffer into a container holding the electrode and thermometer or ATC sensor. Allow the temperature to equilibrate for 1 minute, then discard the used buffer into a waste container.
10. Calibration Range Check.
  - a. Pour a fresh portion of third pH buffer into a container holding the electrode and thermometer or ATC sensor.
  - b. Stir without forming a vortex or swirl slowly (see step 4b).
  - c. Measure the temperature of the buffer solution (remove the liquid-filled thermometer and check the temperature-adjusted pH value), if necessary for the meter being used.
  - d. The pH instrument system should read the value of the third buffer at a known temperature within  $\pm 0.1$  pH units.
    - Meters reading to three or more places to the right of the decimal may not provide better accuracy than  $\pm 0.05$  units, and their accuracy must be verified.

- If it checks, the instrument system is calibrated over a range of pH 4 to 10 and is ready for ANC or alkalinity titrations as well as pH measurement.
- If the instrument system does not check over the entire range, recalibrate before measuring the sample pH. Recalibrate before an alkalinity/ANC titration if the sample has a pH greater than 7.0.

e. Discard the used buffer into a waste container.

f. Rinse the electrode and thermometer (or ATC sensor) with DI water

#### **4.2.2 Measurement**

It is generally preferable to measure pH in situ rather than on a sample taken from a splitter or compositing device. If stream conditions are such that water would pass the in situ pH sensor at a very high rate of flow, however, streaming-potential effects could affect the accuracy of the measurement. For such conditions, it is preferable to withdraw a discrete sample directly from the stream or compositing device and use the sub-sample measurement procedures described below. The pH instrument system should be set up on board the boat or on-shore so that pH is measured at the time of sample collection.

The pH of a water sample can change significantly within hours or even minutes after sample collection as a result of degassing (such as loss of carbon dioxide, hydrogen sulfide, and ammonia); mineral precipitation (such as formation of calcium carbonate); temperature change; and other chemical, physical, and biological reactions. The electrometric method of pH measurement described below applies to filtered or unfiltered surface water and ground water, from fresh to saline.

Field conditions, including rain, wind, cold, dust, and direct sunlight can cause measurement problems. To the extent possible, shield the instrument and measurement process from the effects of harsh weather.

Record the pH variation from a cross-sectional profile, if possible, to determine if pH is uniform at any given discharge, and select the sampling method appropriate for study objectives.

- To sample a shallow flowing stream, wade to the location where pH is to be measured.
- To sample a stream or river too deep to wade, lower a weighted pH sensor with a calibrated temperature sensor (if needed) from a bridge, cableway, or boat. Do not attach the weight to a sensor or sensor cables.
- To sample under still water conditions, measure pH at multiple depths at several points in the cross-section.

**Procedure:**

1. Ensure that the instrument is calibrated.
2. Immerse the pH probe in the water to the correct depth and hold it there for at least 60 seconds to allow for temperature equilibration.
3. Record the pH and temperature values without removing the sensor from the water.
  - Values generally stabilize quickly within  $\pm 0.05$  to 0.1 standard pH unit, depending on the instrument system.
  - Record the median of the observed values.
  - If readings do not stabilize after extending the measurement period, note this on the field forms along with the pH readings, and record the median value of the last five or more readings.
  - After measurements are completed, rinse the pH probe with deionized water and continue with further sampling.
4. For EWI (Equal Width Increment) or EDI (Equal Depth Increment) measurements—Proceed to the next station in the cross section. Repeat steps 3 through 5. After all stations in the cross section have been measured, rinse the sensors with deionized water and store them.
5. Record the mean or median stream pH on the field forms
  - **In still water**—median of three or more sequential values.

- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI**—mean or median of all subsections measured.

### 4.3 SPECIFIC CONDUCTANCE METERS

Specific conductance is used as an indicator of water quality. It is a simple indicator of the change within a system and provides useful information for site characterization. Any meter used to collect field specific conductance measurements will be equipped with a temperature compensator, and read directly in micromhos per centimeter ( $\mu\text{mhos/cm}$ ) corrected to 25°C. The meter will be calibrated to record values over the anticipated range of conductivity values during measurement.

#### 4.3.1 Calibration

Reagent-grade potassium chloride (KCl) will be used for the calibration of specific conductance equipment. Calibration standards will not be used beyond the date specified by the manufacturer. Consult the manufacturer's instruction manual for further details. Specific conductance readings will be reported on the field logs in micromhos/centimeter ( $\mu\text{m/cm}$ ) or millimhos/cm. The instrument calibration will be checked before every water-quality field trip and periodically throughout the sampling event against a standard solution of KCl.

**Procedure:** Calibration and operating procedures differ with instrument systems—always check the manufacturer's instructions.

1. Inspect the instrument and the conductivity sensor for damage. Check the battery voltage. Make sure that all cables are clean and connected properly.
2. Turn the instrument on and allow sufficient time for electronic stabilization.
3. Select the correct instrument calibration scale for expected conductivity.

4. Select the sensor type and the cell constant that will most accurately measure expected conductivity.
5. Select two conductivity standards that will bracket the expected sample conductivity. Verify that the date on the standards has not expired.
6. Equilibrate the standards and the conductivity sensor to the temperature of the sample.
  - a. Put bottles of standards in a minnow bucket, cooler, or large water bath that is being filled with ambient water.
  - b. Allow 15 to 30 minutes for thermal equilibration. Do not allow water to dilute the standard.
7. Rinse the conductivity sensor, the thermometer (liquid-in-glass or thermistor), and a container large enough to hold the dip-type sensor and the thermometer.
  - a. First, rinse the sensor, the thermometer, and the container three times with deionized water.
  - b. Next, rinse the sensor, the thermometer, and the container three times with the standard to be used.
8. Put the sensor and the thermometer into the rinsed container and pour in fresh calibration standard.
9. Measure water temperature. Accurate conductivity measurements depend on accurate temperature measurements or accurate temperature compensation.
  - a. If the sensor contains a calibrated thermistor, use this thermistor to measure water temperature.
  - b. If using a manual instrument without a temperature display or temperature compensation, adjust the instrument to the temperature of the standard using a calibrated liquid-in-glass or a thermistor thermometer.
10. Agitate a submersible-type conductivity sensor up and down under the solution surface to expel air trapped in the sensor. Read the instrument display. Agitate the sensor up and down under the solution surface again, and read the display. Repeat the procedure until consecutive readings are the same.

11. Record the instrument reading and adjust the instrument to the known standard value.

- a. For non temperature-compensating conductivity instruments, apply a temperature-correction factor to convert the instrument reading to conductivity at 25°C.
- b. The correction factor depends to some degree on the specific instrument used—use the temperature-correction factor recommended by the manufacturer.
- c. If an instrument cannot be adjusted to a known calibration standard value, develop a calibration curve. After temperature compensation, if the percentage difference from the standard exceeds 5 percent, refer to the instrument troubleshooting guide.

12. Record in the instrument log book and on field forms:

- a. The temperature of the standard solution.
- b. The known and the measured conductivity of the standard solution (including  $\pm$  variation).
- c. The temperature-correction factor (if necessary).

13. Discard the used standard into a waste container. Thoroughly rinse the sensor, thermometer, and container with deionized water.

14. Repeat steps 7 through 13 with the second conductivity standard.

- a. The purpose for measuring a second standard is to check instrument calibration over the range of the two standards.
- b. The difference from the standard value should not exceed 5 percent.
- c. If the difference is greater than 5 percent, repeat the entire calibration procedure. If the second reading still does not come within 5 percent of standard value, refer to a troubleshooting guide or calibrate a backup instrument.
- d. Switching instrument calibration scales could require re-calibration.

15. Record in the instrument log book and on field forms the calibration data for the second standard.

#### **4.3.2 Measurement**

In situ measurement is preferred for determining conductivity of surface water. Conductivity measurements should be performed immediately upon sample collection at the field site.

Field conditions (rain, wind, cold, dust, direct sunlight) can cause measurement problems—Shield the instrument to the extent possible and perform measurements in a collection chamber in an enclosed vehicle or an on-site laboratory.

For waters susceptible to significant gain and loss of dissolved gases, make the measurement within a gas-impermeable container (Berzelius flask) fitted with a stopper—Place the sensor through the stopper and work quickly to maintain the sample at ambient surface-water or ground-water temperature.

Avoid contamination from the pH electrode filling solution—Measure conductivity on a separate discrete sample from the one used for measuring pH; in a flowthrough chamber, position the conductivity sensor upstream of the pH electrode.

The conductivity measurement reported must account for sample temperature. If using an instrument that does not automatically temperature compensate to 25°C, record the uncompensated measurement in your field notes, along with the corrected conductivity value. Use correction factors supplied by the instrument manufacturer.

Conductivity measurements in flowing surface water should represent the cross-sectional mean or median conductivity at the time of observation. Any deviation from this convention must be documented in the data base and with the published data.

Before beginning, take a cross-sectional conductivity profile to determine the degree of system variability if feasible. A submersible sensor works best for this purpose.

#### **Procedure:**

1. Calibrate the conductivity instrument system at the field site after equilibrating the buffers with stream temperature.



2. Record the conductivity variation from a cross-sectional profile on a field form and select the sampling method.

- Flowing, shallow stream—wade to the location(s) where conductivity is to be measured.
- Stream too deep or swift to wade—lower a weighted conductivity sensor from a bridge, cableway, or boat. Do not attach weight to the sensor or the sensor cable.
- Still-water conditions—measure conductivity at multiple depths at several points in the cross section.

3. Immerse the conductivity and temperature sensors in the water to the correct depth and hold there (no less than 60 seconds) until the sensors equilibrate to water conditions.

4. Record the conductivity and corresponding temperature readings without removing the sensors from the water.

- Values should stabilize quickly to within 5 percent at conductivity  $\leq 100$   $\mu\text{S}/\text{cm}$  and within 3 percent at conductivity  $> 100$   $\mu\text{S}/\text{cm}$ .
- Record the median of the stabilized values on field forms.
- If the readings do not meet the stability criterion after extending the measurement period, record this difficulty in the field notes along with the fluctuation range and the median value of the last five or more readings.

5. For EWI or EDI measurements, proceed to the next station in the cross section and repeat steps 3 and 4. Record on field forms the mean (or median, if appropriate) value for each subsection measured.

6. When the measurement is complete, remove the sensor from the water, rinse it with deionized water, and store it.

7. Record the stream conductivity on the field forms:

- **In still water**—median of three or more sequential values.

- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI**—mean or median of all subsections measured (see NFM 6.0).

### Sub-sample measurement

Representative samples are to be collected and split or composited according to approved USGS methods. Measure the conductivity of samples as soon as possible after collection. If the sample cannot be analyzed immediately, fill a bottle to the top, close it tightly, and maintain the sample at stream temperature until measurement.

Reported conductivity values normally are determined on an unfiltered sample. Large concentrations of suspended sediment can be a source of measurement error—record such conditions in the field notes.

- If sediment concentrations are heavy, measure conductivity on both unfiltered and filtered sub-samples and record both values on the field form.
- If the conductivity value differs significantly between the filtered and unfiltered samples, report the filtered value as sample conductivity and identify it as a “filtered sample.”

1. Calibrate the conductivity instrument system at the field site.
2. Select the sampling method (see NFM 6.0) and collect a representative sample.
3. Withdraw a homogenized sub-sample from a sample splitter or compositing device. Rinse the sample bottles three times with the sample—rinse them with sample filtrate, for filtered samples.
4. Rinse the conductivity sensor, the thermometer (liquid-in-glass or thermistor), and a container large enough to hold the dip-type sensor and the thermometer.
  - a. First, rinse the sensor, the thermometer, and the container three times with deionized water.

b. Next, rinse the sensor, the thermometer, and the container using sample water.

5. Allow the sensors to equilibrate to sample temperature, then discard the used sample water. Pour fresh sample water into a container holding the sensor and the thermometer. When using a dip-type sensor, do not let the sensor touch the bottom or sides of the measuring container.

6. Measure water temperature.

- If the conductivity sensor contains a calibrated thermistor, use this thermistor to measure water temperature.
- If the instrument is not temperature compensating, use a calibrated thermistor or a liquid-in-glass thermometer.
- Adjust the instrument to the sample temperature (if necessary) and remove the thermometer.

7. Measure conductivity.

- a. Remove any air trapped in the sensor by agitating the sensor up and down under the water surface.
- b. Read the instrument display.
- c. Agitate the sensor up and down under the water surface, and read the display again.
- d. Repeat the procedure until consecutive readings are the same.

8. Record the conductivity and the sample temperature on field forms.

- If the instrument is not temperature compensating, record the raw data and convert the values to conductivity at 25°C using temperature-correction factors provided by the manufacturer.
- Report the median of the readings to three significant figures on the field forms.
- Discard the sample into a waste container and dispose according to regulations.

#### 9. Quality control-

- Repeat steps 3 through 8 with at least two fresh subsamples, rinsing the instruments once only with sample water.
- Subsample values should be within  $\pm 5$  percent for conductivity  $\leq 100$   $\mu\text{S/cm}$ , or  $\pm 3$  percent for conductivity  $> 100$   $\mu\text{S/cm}$ .
- If criteria cannot be met: filter the samples, report the median of 3 or more samples, and record this difficulty in field notes.

10. Rinse the sensor, the thermometer, and the container with deionized water. If another measurement is to be made within the next day or two, store the sensor in deionized water. Otherwise, store the sensor dry.

### 4.4 TURBIDITY METERS

Turbidity meters measure the amount of light scattered at right angles from a beam of light passing through the test sample. Turbidity readings are the measure of the interaction of light with suspended solid particles in the sample. Test results are read directly in Nephelometric Turbidity Units (NTUs) on an LCD digital readout.

#### ***4.4.1 Calibration***

The turbidity meter is pre-calibrated in the factory, and a simple standardization is the only step required prior to testing.

#### ***4.4.2 Measurement***

Turbidity measurements should be repeated three to five times to ensure accuracy and replication within the precision of the instrument.

Benchtop determination of turbidity is especially susceptible to negative bias from particle settling. Visually check for the presence of coarse material (sand or coarse silt) in the sample. Gently agitate the sample, then set it down. If particles rapidly settle to the

bottom (within 3-5 seconds), then coarse materials are present and the sample cannot be measured accurately using the static method. Static measurements made on such samples therefore must be coded to indicate that accuracy is qualified when being entered into a database.

**Procedure:**

***Preliminary steps for benchtop turbidity determination:***

1. Warm up the turbidimeter according to the manufacturer's instructions. Put on powderless laboratory gloves.
2. Rinse a clean, dry, scratch-free, index marked cell with a turbidity calibrant within the range of interest.
3. Gently agitate the calibrant, pour the calibrant into the sample cell to the fill mark, and dry the cell exterior with a lint-free cloth. When using a meter recently calibrated with an acceptable calibrant turbidity solution (formazin or styrene-divinylbenzene polymer), a verification calibrant may be used for this check measurement.
4. Follow the manufacturer's instructions for readout of turbidity value and record the turbidity of the calibrant used and the turbidity value measured in the calibration logbook. If readings are not within specifications for the indicated range, recalibrate the instrument for the turbidimeter using accepted calibration turbidity solutions.

***For samples with turbidity less than 40 turbidity units:***

1. Measure sample turbidity immediately or as soon as possible upon sample withdrawal.
  - a. If discrete sub-samples are to be taken from a churn splitter or other sample-compositing device, remove samples for turbidity measurement along with other whole water samples. Avoid pouring the sample into a cuvette from a bottle, if possible. If not possible, then invert the bottle 25 times using a 1-second inversion cycle and pour off the sample immediately to capture suspended particles.

- b. For drinking water, use an instrument that complies with EPA Method 180.1 or GLI Method 2. Measurements are reported in NTU or NTRU for EPA 180.1, or in FNMU for GLI Method 2.
2. Rinse a freshly cleaned cell with the sample to be tested.
3. For a discrete (static) sample, complete the following sequence of steps (through step 4a) without hesitation (skip to step 4 for flowthrough cell measurement).
  - a. Gently invert—do not shake—the sample 25 times to completely disperse the solids, taking care not to entrain air bubbles. Allow air bubbles to disappear before filling the sample cell.
  - b. Rapidly pour the sample into a sample cell to the line marked (to the neck if there is no line). Do not touch cell walls with fingers.
  - c. Remove condensation from the cell with a clean, soft, lint-free cloth or tissue. If condensation continues, apply a thin coating of silicon oil to the outside of the cell about every third time the cell is wiped dry of moisture. Allow samples to equilibrate to ambient temperature, if necessary, before sub-sampling to help minimize condensation problems. Note: warming the sample may change particle associations in the water matrix.
  - d. Before inserting the sample cell into the meter, ensure that no air bubbles are present in the cell. If necessary, degas the sample according to the manufacturer's instructions. Air bubbles can cause significant positive bias in turbidity measurements.
  - e. Orient the calibration cell in the cell holder according to the index marks—the calibration cell and sample cell must have identical orientation when in the instrument measurement chamber.
4. Determine the measured turbidity value of the sample directly from the instrument scale or by using the instrument value and calibration curve, as is appropriate for the

instrument being used. For samples with less than 1 turbidity unit, see the Technical Note under step 4d.

- a. Record the very first readings after placement of the sample cell in the measurement chamber. If readings are unstable, then particle settling may be occurring. If so, gently re-invert the cell 25 times and record at least three readings over a short, defined time interval (for example, 30 seconds to 1 minute).
- b. Repeat at least twice with fresh sample, until three or more sample values fall within  $\pm 10$  percent.
- c. Samples that contain significant color should be diluted if using EPA Method 180.1 (for samples with turbidity greater than 40 units see below "For samples, including drinking water, with turbidity greater than 40 turbidity units," step 3). Results of diluted samples must be qualified with a "d" in the "Value Qualifier Code" field for data entered into the USGS NWIS database.
- d. Report the median of the three or more sequential readings that fall within  $\pm 10$  percent.

**Technical Note:** When using low-level reporting scales, you may need to subtract a correction factor from the reading to correct for stray light. For example, the Hach Company reports the correction for the 0.2-NTU scale to be on the order of 0.04 NTU for the Hach 2100P. The stray-light correction is determined by reading turbidity from an empty instrument (without cuvette).

5. Record the data. If particle settling or instability in initial readings was a problem, add documentation to field notes in the log book.

***For samples, including drinking water, with turbidity greater than 40 turbidity units:***

1. Select an appropriate instrument.

- For drinking water, use EPA Method 180.1, a compliant instrument, and NTU or NTRU reporting units; alternatively, select the GLI Method 2, a compliant instrument, and FNMU reporting units. Reporting units for these methods must be remarked with an “E” code in NWIS for turbidities greater than 40.
- For study objectives other than drinking water, choose instruments according to study objectives.

2. Obtain a discrete sample.

- For drinking-water samples, proceed to step 3.
- For non-drinking-water samples, skip to step 4.

3. For drinking-water samples, dilution is required to comply with USEPA regulations.

- a. Dilute the sample with one or more equal volumes of turbidity-free water until turbidity is less than 40 turbidity units after mixing and degassing.
- b. Record the volume of turbidity-free water used for dilution. Follow steps 1-5 from the previous section for samples with turbidity less than 40 turbidity units.
- c. Skip to step 5, below

4. For non-drinking-water samples (where USEPA compliance is not required), with 100 and 1,000 turbidity-unit ranges only — place a cell riser (if available) into the cell holder before inserting the sample cell. This decreases the length of the light path in order to improve the linearity of measurements. Do not use the cell riser for the lower turbidity ranges.

- a. For turbidimeters with adjustable ranges and signal-processing capabilities (for instance, ratio mode to compensate for high particle densities), select the desired configuration and operate according to manufacturer’s recommendations. Some instruments will automatically switch to different modes (for example, ratio mode) or to a different light source. Record instrument mode on field sheets.
- b. Select the desired range on the turbidimeter.

5. Fill the cell with sample water:

- a. Hold the cell by the rim (top lip), not beneath the lip.



- b. Gently agitate the sample 25 times. Without hesitation, carefully but rapidly pour sample water into the cell to the fill mark.
- c. Wipe the exterior of the cell using a soft, lint-free cloth or tissue to remove moisture (condensation) from cell walls.
- d. If necessary, apply a thin layer of silicon oil onto the exterior of the cell to reduce condensation on the cell and mask slight scratches and nicks.
- e. If rapid particle settling is occurring, steadily invert the cell 25 times, taking care not to shake too vigorously, which could entrain gases in the sample.

6. Record the sample turbidity.

Most modern turbidimeters will adjust initial sample readings directly into a final reading based on the previous calibration. If the meter does not have this capability, you will need to read values from a calibration curve constructed previously.

- a. Record the very first readings after placement of the sample cell in the measurement chamber. If readings are unstable, particle settling may be occurring: gently re-invert the cell 25 times and record at least three readings over a defined time interval (for example, 30 seconds to 1 minute).
- b. Repeat at least twice with fresh sample until three or more sample values fall within  $\pm 10$  percent.
- c. Samples that contain significant color should be diluted if using EPA Method 180.1. Results of diluted samples must be qualified with a "d" in the "Value Qualifier Code" field for data entered into the USGS NWIS database.
- d. Report the median of the three or more sequential readings that fall within  $\pm 10$  percent.
  - For diluted water samples, the measured turbidity must be converted based on the amount of dilution, according to the following equation:

$$T_s = T_d \times (V_o + V_s) / V_s$$

where  $T_s$  = turbidity of the environmental sample,  $T_d$  = turbidity of the diluted sample,  $V_o$  = volume of turbidity-free water in the diluted mixture, and  $V_s$  = volume of the environmental sample in the diluted mixture.

**EXAMPLE:** If five volumes of turbidity-free water were added to one volume of sample, and the diluted sample showed a turbidity of 30 units, then the turbidity of the original sample is computed as 180 units.

e. Report turbidity as follows, using method codes as described in [http://water.usgs.gov/owq/turbidity\\_codes.xls](http://water.usgs.gov/owq/turbidity_codes.xls):

- For EPA Method 180.1, use NTU or NTRU.
- For GLI Method 2, use FNMU.
- For non-diluted, non-USEPA-compliant measurements, use the appropriate reporting units.

## 4.5 DISSOLVED OXYGEN METERS

Dissolved oxygen (DO) meters measure the quantity of oxygen dissolved in water. In a typical DO meter, the tip of the probe consists of a cell enclosed by a selective membrane in a protective holder containing the electrolyte and electrodes.

### 4.5.1 Calibration

Always calibrate the instrument according to the manufacturer's specifications. For an accurate calibration, the probe may require immersion in water in an airtight container. If an open container is used for calibration, the margin of error is approximately 0.1 ppm. If the calibration is performed above sea level, a correction will be made for the difference in altitude. Certain table listings are available for oxygen solubility as a function of temperature and salinity. Refer to the manufacturer's specifications regarding slope calibration.

Instrument systems for the amperometric or the luminescent-sensor methods must be properly calibrated and tested before each field trip and cleaned in the field after each use.

- *Amperometric instruments*

Different manufacturers recommend different calibration frequencies for membrane-electrode DO meters; however, virtually all state that optimum instrument performance and data quality will be obtained by frequent calibration. Calibration and operation procedures for the amperometric method differ among instrument types and makes—refer to the manufacturer's instructions.

- *Luminescent-sensor instruments*

Luminescent-based sensors are pre-calibrated by the manufacturer and most manufacturers' literature suggests that no further calibration is warranted. The accuracy of factory calibrations, however, may not satisfy the data-quality objectives of a specific program. Frequency of calibration can have a significant effect on the overall accuracy and precision of DO measurements; therefore, users of these meters are advised to make frequent calibration checks and to recalibrate as frequently as required to meet specific data-quality objectives.

- *One-point and two-point calibrations*

Calibration for most amperometric DO instruments and some luminescent-sensor instruments can only be checked with a 1-point calibration at 100-percent saturation. For these instruments, a zero DO check should be performed routinely as an evaluation of sensor performance. Because the sensors on DO instruments may be slow to respond after the zero check, the sensor should be thoroughly rinsed with deionized water before use.

Some instruments allow for 2-point calibrations at 0-percent and 100-percent saturation. Follow the manufacturer's instructions for those instruments with 2-point calibration functionality. Verifying instrument performance at zero DO and using a 2-point calibration can be particularly important for data accuracy when the instrument will be used to measure low DO concentrations (less than 5 mg/L).

- *Correction for atmospheric pressure*

Atmospheric pressure, the temperature of the water or water vapor, and the conductivity (or salinity) of the water must be known to determine the theoretical amount of oxygen that can be dissolved in water. Record all calibration information in instrument log books and copy calibration data onto field forms at the time of calibration.

Ambient atmospheric pressure is true atmospheric pressure at the measurement site, not that which has been adjusted to sea level. Atmospheric pressure reported by the National Weather Service generally is not the true (ambient) value. Weather Service atmospheric readings usually are adjusted to sea level and must be adjusted back to the elevation of the weather station. Upon request, a weather station may provide ambient atmospheric pressure.

- Use a calibration-checked pocket altimeter-barometer to determine ambient atmospheric pressure to the nearest 1 millimeter (mm) of mercury.
- Check the accuracy of all field barometers before each field trip, and record readings and adjustments in the log book. If possible, check barometer accuracy with information from an official weather station.
- Use Table 1 and Figure 1 if the value used for atmospheric pressure has been adjusted to sea level.
- To correct weather station readings adjusted to sea level to ambient atmospheric pressure: subtract appropriate values shown (Table 1, Figure 1) from atmospheric readings adjusted to sea level (shown in millimeters of mercury).

Although atmospheric pressure does not decrease linearly with increases in elevation, linear interpolation is acceptable within the elevation ranges given in Table 1. Alternatively, plot the values from Table 1 and extrapolate subtraction factors directly from the graph (Figure 1). Many instruments have the pressure-temperature algorithm stored in internal memory. Interactive tables also are available for user-specified temperature, pressure, and salinity at <http://water.usgs.gov/software/dotables.html>.

Table 1. Factors used to correct atmospheric pressures adjusted at sea level  
NGVD, National Geodetic Vertical Datum of 1929

Elevation of weather station (in feet, NGVD)	Value to subtract (mm of Hg)
0	0
1000	27
2000	53
3000	79
4000	104
5000	128
6000	151

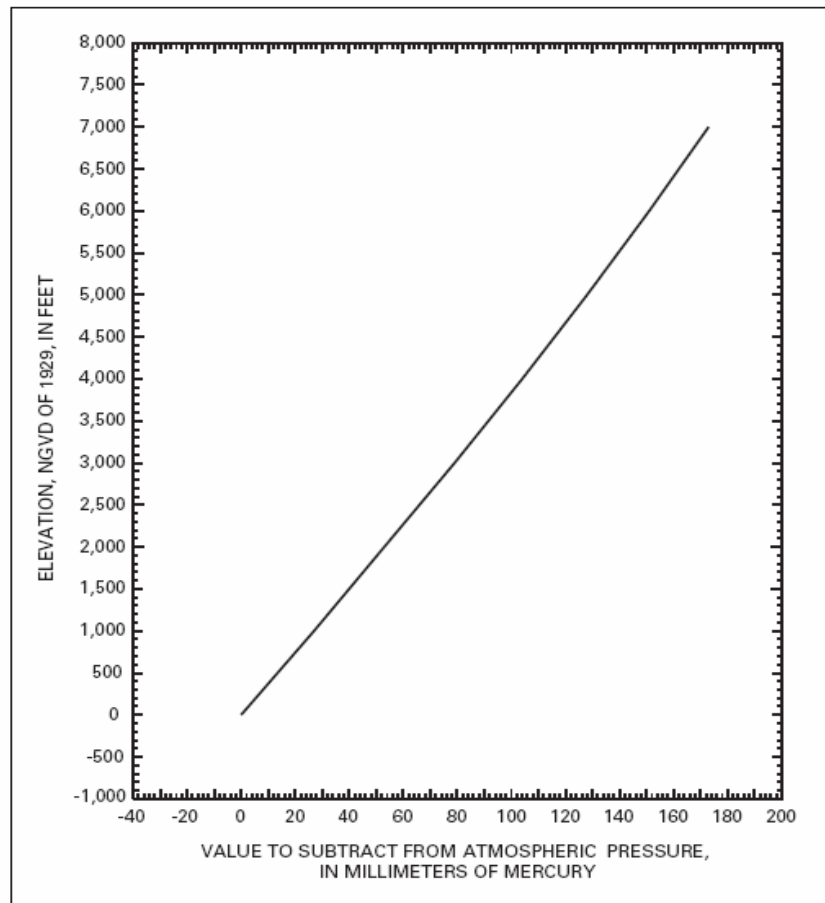


Figure 1. Factors used to correct atmospheric pressures adjusted to sea level.

***Procedure 1—Air-calibration chamber in air***

This calibration method is most commonly recommended by manufacturers of amperometric instruments. Calibration chambers are either built into the instrument case or are provided as separate components by the manufacturer. Use the calibration chamber provided or recommended by the manufacturer.

1. Wet the inside of the calibration chamber with water. Then pour out the water (but leave a few drops). Remove any water droplets on the sensor membrane and insert the sensor into the chamber (this ensures 100-percent humidity).
2. If using an amperometric instrument, allow 10 to 15 minutes for the DO sensor and the air inside the calibration chamber to equilibrate.
3. Using your calibration pocket altimeter-barometer, read the ambient atmospheric pressure checked to the nearest 1 mm of mercury.
4. Measure the temperature in the calibration chamber and observe the readings until the instrument stabilizes. Read the temperature to the nearest 0.1°C. The temperature inside the chamber should approximate the water temperature, measured with a calibrated thermometer.

**Technical Note for Amperometric Instruments:** Most instrument manufacturers recommend calibrating at temperatures that are at least within 10°C of the ambient water temperature. The most accurate calibration will be achieved if the temperature difference between the environmental water and the calibration chamber is minimized as much as possible.

5. Use Table 1 to determine the DO saturation at the measured temperature and atmospheric pressure.
6. Following the manufacturer's instructions, adjust the calibration control until the instrument reads the DO saturation value determined from Table 1. Verify that the instrument reading is within  $\pm 0.2$  mg/L of the computed saturation value, or use more stringent accuracy criteria that reflect the data-quality requirements of the study. The luminescent-sensor instrument is now calibrated and ready for use.

7. When working with an amperometric instrument, remove the sensor from the calibration chamber and check to see if any water droplets are on the membrane. Water droplets on the membrane cause improper calibration. If water droplets are present, recalibrate the instrument; otherwise the instrument is now calibrated and ready for use.

***Procedure 2—Calibration with air-saturated water***

In this procedure, the DO sensor or instrument system is calibrated against water that is saturated with oxygen at a known temperature and ambient atmospheric pressure.

1. The temperature of water used for calibration should be about the same as the temperature and conductivity of the water to be measured.

- **If working at the field site**—obtain about 1 liter (L) of water from the water body to be measured.
- **If working in the laboratory**—obtain about 1 L of deionized water or tap water.

2. Place the DO sensor and calibration water in a large beaker or open-mouth container. (Some manufacturers supply an air-saturated water-calibration vessel.)

- Allow the sensor to come to thermal equilibrium with the water temperature.
- Shield the beaker or container from direct sunlight and wind to minimize temperature variations.

3. Aerate the water for 5 to 10 minutes. Using a battery-operated aquarium pump or minnow-bucket aerator and a short piece of tubing, attach a gas diffusion stone to the end of the tubing and place it at the bottom of the beaker of calibration water. Avoid placing the instrument in the stream of air bubbles.

4. Determine if the water is 100-percent saturated with oxygen.

- Observe the instrument reading while aerating the calibration water.
- When no change in the DO reading is observed on the instrument for 4 to 5 minutes, assume that the water is saturated.

5. Using your pocket altimeter-barometer, read the ambient atmospheric pressure to the nearest 1 mm of mercury.

6. Read the temperature of the calibration water to the nearest 0.1°C.
7. Using oxygen solubility Table 1, determine the DO saturation value at the measured temperature and atmospheric pressure of the calibration water.

**\*\*\*Skip to Step 9 if using an amperometric instrument\*\*\***

8. **For luminescent-sensor instruments:** Following the manufacturer's instrument calibration instructions, verify that the instrument reading is within  $\pm 0.2$  mg/L of the computed saturation value. (Alternatively, apply a more stringent accuracy criterion that reflects study data-quality requirements.) **The luminescent-sensor instrument is now calibrated and ready for use.**

9. **For amperometric instruments:** Adequate flow of water across the surface of the membrane is required for accurate measurements. Recommendations for flow velocity vary by manufacturer, with most recommending about 1 foot per second (ft/s).

- Provide suitable turbulence in the air-saturated water by physical or mechanical means to maintain the required flow rate past the membrane, avoiding the creation of air bubbles at the water-sensor interface.
- Maintain this flow rate when making measurements and adjusting instrument calibration.

10. **For amperometric instruments:** Turn off the aerator and take care to prevent any air bubbles from adhering to the membrane. Following the manufacturer's instructions, set or adjust the calibration control until the instrument reads a saturation value of DO as determined above. Verify that the instrument reading is within  $\pm 0.2$  mg/L of the computed saturation value, or use more stringent accuracy criteria that reflect the data-quality objectives of the study.

### ***Procedure 3—Air-calibration chamber in water***

**This calibration method is applicable only to amperometric instruments.** An air-calibration chamber permits calibration of the DO sensor at the temperature of the water in which the DO concentration is to be measured. This calibration procedure minimizes errors caused by temperature differences. Air-calibration chambers for in-water



calibrations currently are not available on the open market and one of the most common, the YSI 5075A calibration chamber, is no longer manufactured. For most multi-parameter water-quality instruments, the manufacturer-provided ground-water flow cell may be modified and used as an air-calibration chamber in water. The modification requires the cell to be mounted on the sonde with one port of the cell plugged and the other port vented to the atmosphere with tubing.

1. Insert the sensor probe into the rings of the DO wand and dip this calibration chamber into the surface or ground water to be measured, allowing the temperature readings to stabilize. Remove the wand and pour out the excess water, leaving a few drops.

- Check for and remove any water droplets on the sensor membrane.
- Insert the DO sensor into the wet chamber (this ensures 100 percent humidity).
- If a YSI model 5739 sensor is used, the pressure-compensating diaphragm on the side of the sensor must be enclosed within the calibration chamber during calibration.
- Check that no water can leak into the calibration chamber and that the membrane does not have droplets of water adhering to it. The water droplets reduce the rate of oxygen diffusion through a membrane, producing erroneous results.

2. Immerse the calibration chamber into the water to be measured. Allow 10 to 15 minutes for the air temperature inside the chamber to equilibrate with the water (see the **Technical Note** in Procedure 1).

- For streams, choose an area of the stream that closely approximates mean stream temperature. In shallow streams, try to place the chamber in an area that represents the stream but that is shaded from direct sunlight.
- For ground water, use temperature-stabilized purge water or other clean water having a temperature that closely approximates that of the ground water.

3. Using a calibration-checked pocket altimeter-barometer, determine the ambient atmospheric pressure to the nearest 1 mm of mercury.

4. Read the temperature within the chamber to the nearest 0.1°C, using a calibrated thermometer.

- The temperature inside the chamber should approximate the water temperature.
- If the two temperatures do not match, allow additional time for equilibration of the chamber with the water temperature.
- If the temperature of the chamber still does not approximate the water temperature, the thermistor in the DO sensor might be malfunctioning. Compare water temperature measured by the DO meter and a calibrated field thermometer. If the two measurements vary by more than  $\pm 0.2^{\circ}\text{C}$ , the calibration should be discontinued and the DO meter thermistor should be repaired following the manufacturer's recommendations.

**Technical Note:** Most instrument manufacturers recommend calibrating at temperatures that are at least within 10°C of the ambient water temperature. The most accurate calibration will be achieved if the temperature difference between the environmental water and the calibration chamber is minimized as much as possible.

5. Use Table 1 to determine the DO saturation value at the measured water temperature and atmospheric pressure.

6. Following the manufacturer's instructions, set or adjust the calibration control until the instrument reads a DO saturation value determined from oxygen solubility (Table 1). Verify that the instrument reading is within 0.2 mg/L of the computed saturation value, or use more stringent accuracy criteria per the data-quality objectives of the study. The instrument is now calibrated and ready for use. Remove the sensor from the calibration chamber.

#### **4.5.2 Measurement**

The solubility of oxygen in water depends on the partial pressure of oxygen in air, the temperature of the water, and the dissolved-solids content of the water.

Standard determinations of dissolved oxygen in surface water represent the cross-sectional median or mean concentration of dissolved oxygen at the time of observation.

Measuring the DO concentration at one distinct spot in a cross section is valid only for flowing water with a cross-sectional DO variation of less than 0.5 mg/L. Discerning such variation requires a cursory cross-section measurement. The effort involved in collecting this cross-section information is only slightly less than making an equal-width-increment (EWI), equal-discharge-increment (EDI), or multiple-vertical cross-sectional measurement. Measurements made at multiple locations in the cross section are recommended when possible.

- Determining DO for a single vertical at the centroid of flow at the midpoint of the vertical only represents the cross section under ideal mixing conditions.
- Do not measure DO in or directly below sections with turbulent flow, in still water, or from the bank, unless these conditions represent most of the reach or are required by the study objectives.
- Apply a salinity correction to the saturation values after the DO measurement, if needed

Dissolved oxygen must be measured in situ. Never measure DO from a sample splitter.

**Procedure:**

1. Calibrate the DO instrument at the field site and check that the temperature thermistor has been certified by the USGS Water Science Center within the past 4 months
2. Record the DO variation from the cross-sectional profile and select the sampling method
  - **Flowing, shallow stream**—Wade to the location(s) where DO is to be measured.
  - **Stream too deep or swift to wade**—Lower a weighted DO sensor with a calibrated temperature sensor from a bridge, cableway, or boat. (Do not attach the weight directly to the sensors or sensor cables, because this could damage the sensors or sensor cables.)
  - **Still-water conditions**—Measure DO at multiple depths at several points in the cross section.
3. Immerse the DO and temperature sensors directly into the water body and allow the sensors to equilibrate to the water temperature (no less than 60 seconds).

**Notes for amperometric instruments only:**

If the water velocity at the point of measurement is less than about 1 ft/s, use a stirring device or stir by hand to increase the velocity. (To hand stir, raise and lower the sensor at a rate of about 1 ft/s, but do not break the surface of the water.) The stir-by-hand method may not be appropriate in lakes, reservoirs, or slow-moving waters (for example, bayous) as these water bodies may be stratified at the point of measurement, making accurate DO measurements impossible. This could be especially problematic in areas where DO

concentrations change substantially over short distances, such as near the thermocline or bottom sediments.

- High stream velocity can cause erroneous DO measurements.

4. Record the temperature without removing the sensor from the water.

5. After the instrument reading has stabilized, record the median DO concentration

The reading should stabilize to within  $\pm 0.2$  mg/L.

6. For EWI, EDI, or multiple-vertical measurements, proceed to the next station in the cross section and repeat steps 3 through 5. When measurements for the stream have been completed, remove the sensor from the water, rinse it with deionized water, and store it according to the manufacturer's instructions.

7. Record DO concentrations on the field forms:

- **In still water**—median of three or more sequential values.
- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI—mean (or median)** of all subsections measured.

## 5.0 QUALITY ASSURANCE AND QUALITY CONTROL

All equipment calibration data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP or manufacturer's recommendations. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

## 6.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the calibration process shall be managed in accordance with the procedures outlined in SOP-12.

## 7.0 REFERENCES

Beta Technology Incorporated. Hydac Instruction Manual for the Hydac Conductivity Temperature pH Tester, Beta Technology Incorporated, 545 Moose Lodge Road, Cambridge, MD 21613-0265.

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LIBBY SUPERFUND SITE OPERABLE UNIT 3 STANDARD OPERATING PROCEDURE

Libby Site OU3 SOP No. 11

Rev. No. 1

Date: January 31, 2008

Page 1 of 7

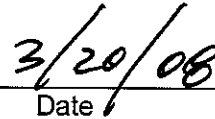
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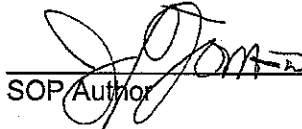
OU3 SOP 11 (Rev 1)

**Title: GPS DATA COLLECTION**

**APPROVALS:**

  
EPA Remedial Project Manager

  
Date

  
SOP Author

  
Date

Revision Number	Date	Reason for Revision
0	9/26/07	
1	1/31/08	Update based on field team input



**LIBBY ASBESTOS SUPERFUND SITE OU3**  
**STANDARD OPERATING PROCEDURE No. 11**  
**GPS DATA COLLECTION**

**1.0 INTRODUCTION**

This Standard Operating Procedure (SOP) is a general guidance document for the collection of coordinates of point locations using a Global Positioning System (GPS) unit. The GPS is a worldwide, satellite-based system with location positioning capabilities. The system is administered and managed by the Department of Defense. It is comprised of:

- a space segment of approximately 24 operational satellites in complimentary orbit,
- a ground control segment made up of a network of control stations around the globe, and
- a user segment, which includes anyone who uses GPS to collect locational information.

The system utilizes precise time and radio signals to determine distances from satellites to user GPS receivers. Distances are most commonly calculated by using the time it takes for a radio signal code to be transmitted from the satellite and received by the GPS unit. Precise time is critical to the successful operation of the system. The control stations ensure that the satellites employ synchronized, atomic clock-derived universal time coordinates (UTC), commonly known as Greenwich Mean Time (GMT). Receiver units collecting four satellite signals can determine the geodetic (x, y, z) location through a process of mathematical triangulation. The satellite signals contain precise time and satellite position information.

GPS technology is used as a method of accurately determining the coordinates of point locations. The three-dimensional position, or the x, y, and z geodetic coordinates, are determined for the point locations; however, only the x and y values are primarily used. This is due to the processes involved in the system; the vertical GPS coordinates are approximately

half as accurate as the horizontal GPS coordinates. The position reported by the GPS unit is based on the geodetic model selected. The vertical, or z coordinate, value is not as accurate as the reported position due to the geometry of the satellite constellation relative to the receiver's position on the earth.

GPS is one of the arrays of tools for accurately determining location in the field. The collection of x, y, and z coordinates (for gross data collection) for locations in the field using GPS is useful for a variety of purposes, including accurate sample locations, locational correlation of remotely sensed data with ground truth locations, and efficiently collecting better spatial data.

### 3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Selects project-specific field documentation with input from other key project staff, and appropriate oversight agencies.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer:** Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

**Field Technician (or other designated personnel):** Assists the FTL and/or field geologist, hydrogeologist, or engineer in the implementation of field tasks and field documentation.

## 4.0 PROCEDURES

There are three major types of GPS units available: survey-grade units, mapping-grade units, and recreational-grade units. For the collection of environmental/natural resource data, mapping-grade and recreational-grade units are usually sufficient. The type of GPS unit employed should meet the data collection needs as outlined in the project work plan.

Recreational-grade GPS units can be used to acquire location information (generally points) when spatial accuracy is not paramount to the project. Recreational GPS units do not have data dictionaries for storing attribute information with the point location. The procedures described here are geared toward hand-held recreational GPS units. Consult the specific instrument's instruction manual for details on operation.

There has been, and will continue to be, a considerable and rapid evolution in GPS techniques and technologies. Adjustments to the following operational procedures may be necessary to reflect these rapid changes in technology.

### 4.1 Method

#### Planning

If a recreational-grade GPS meets the criteria of the project, the unit chosen must have the capability of downloading collected data to a personal computer. This is usually accomplished with a parallel or USB cable connection.

Much of the data collected by GPS will eventually reside in a relational database. Each GPS feature collected should contain a unique identifier that relates the feature to an associated record in a database. Since recreational GPS units have only one text field for input, careful consideration should be given to the use of this field and the design of unique identifiers.

#### Data Collection

Locational data are captured by recreational-grade GPS units as *waypoints*. When taking a waypoint, enter the Location ID in the text field provided. It is also recommended that reference points be collected occasionally. These reference point positions should be taken at known

locations (e.g., site headquarters office, stream confluences) which can later be used in GIS to QC the accuracy of waypoint data.

If navigation to preset waypoints is applicable to a project, they must be loaded onto the GPS unit before departure to the field. It is also recommended to have printed topographic maps of the waypoint locations in order to maximize field time and efficiently navigate between waypoints.

### Data Processing

GPS units should be downloaded once a day or after each field session. Data should be downloaded both as a text file and a shapefile. Points should be checked for reasonable spatial accuracy and errors. Subsequent downloads should be error-checked in the same manner. When data collection is finished, all files should be compiled into one spatial file.

### Additional Documentation

Regardless of the type of GPS unit used to collect locational data, all resulting GIS datasets need to have information documenting how the GPS data were collected. Documentation can be recorded at the time of data point collection and/or can be stored along side the electronic data set with a simple readme text file.

The following details are suggested as items to include in data collection:

- Name of project
- Name(s) of data collectors
- Coordinate system (projection, datum & zone)
- Type (or types) of GPS units used
- The range of field collection dates

Below is an example of typical GPS data collection using a hand-held recreational-use GPS device. It serves only as an example of data collection and is not intended to provide detailed step-by-step instructions operation. Always refer to the owner's manual for specific instructions on device operation and data collection.

- Turn GPS unit on by pressing and releasing the power key.
- Following the Welcome Page the Satellite Page will be next.
- After sufficient satellites have been acquired, change to Position Page.
- When locating a sample location, use the sample identification as described in the field sampling plan.
- To record a location press the Mark key; the longitude, latitude, time, and date will be saved. Record the information into a field log book, and save the information in the GPS unit with a unique identification name and/or number to be downloaded later.
- Then enter OK to return to the Position Page.
- To turn off the GPS unit press and hold down the power key.

## **5.0 QUALITY ASSURANCE AND QUALITY CONTROL**

Only data with high geometrical strength or low percent dilution of position (PDOP) will be used to ensure high accuracy. The Field Team Leader or designated QA reviewer will check and verify that the GPS coordinates are collected using the appropriate Datum, are entered into a field logbook or electronic database on a daily basis and that coordinates entered into project records match those recorded in the GPS-unit memory. If any corrections are necessary, the field team lead or other field personnel will make those corrections before coordinates are transmitted to data users.

All GPS equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

## 6.0 REFERENCES

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United States National Park Service. 2004. "GPS Field Data Collection Guide, Appendix H. NCPN Specifications for Using Global Positioning Systems." Version 1.1. December. 12 pgs. [http://science.nature.nps.gov/im/units/ncpn/link\\_library/NCPN\\_GPS\\_guidelines\\_Appendix\\_H.pdf](http://science.nature.nps.gov/im/units/ncpn/link_library/NCPN_GPS_guidelines_Appendix_H.pdf)

Garmin GPSmap 60CSx Operators Manual

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007

OU3 SOP 12 (Rev. 0)

Title: INVESTIGATION DERIVED WASTE (IDW) MANAGEMENT

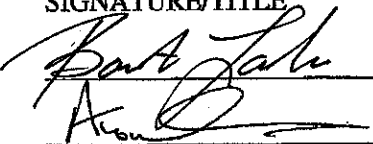
APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

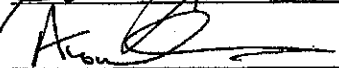
DATE

EPA Remedial Project Manager



9/26/07

SOP Author



9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--



## 1.0 INTRODUCTION

This SOP is based on MWH SOP-07, Investigation-Derived Waste (IDW) Management, Revision 1.0, April 2007, modified for use at the Libby Asbestos Superfund Site OU3. IDW may be generated during field investigations at the Libby Asbestos Superfund Site OU3. The National Contingency Plan (NCP), codified in 40 Code of Federal Regulations (CFR) 300, requires that IDW be handled to attain all the applicable or relevant and appropriate requirements (ARARs) to the extent practicable, considering the urgency of the situation. The purpose of this SOP is to present procedures to be followed in the management of IDW generated during the field investigations.

Typical IDW generated during field activities are solid wastes and may include (but are not limited to) the following media and waste types:

Fluids	Solids
Purge water and groundwater	Soils and soil cuttings
Drilling mud	Plastic tarps or sheeting
Grout	Drill pipe and well casing/screen
Decontamination fluids and wastewater	Decontamination solids
	Disposable equipment (i.e., rope, bailers, sampling equipment, & other consumables)
	Spent personal protective equipment (PPE)
	Used containers, sample bottles
	Packaging materials

The above wastes may or may not be encountered, generated or managed while performing field investigations. However, all solid waste streams will be characterized to determine if they are hazardous wastes per 40 CFR § 262.11 for the purposes of handling and disposal. Guidance from this document shall be used as part of project planning to estimate total volumes of IDW likely to be generated as well as how the IDW will be managed and disposed.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in IDW handling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## 3.0 DEFINITIONS

**Area of Contamination (AOC) unit:** The AOC unit concept is critical to the IDW management at a CERCLA investigation site. Although EPA has not promulgated a definition of an AOC unit, an AOC unit is generally an area within a CERCLA investigation site with similar characteristics with respect to contamination and the associated risks to human health and the environment. A CERCLA investigation site may contain one or more AOC units.

**Decontamination fluids:** Any fluids, including aqueous wash water, solvents, and contaminants that are used or generated during decontamination procedures.

**Decontamination solids:** Any solids, including soils and soil cuttings, fill materials, and contaminants that are generated during decontamination procedures.

**Grout:** A fluid mixture of cement and water (neat cement) of a consistency that can be forced through a pipe and placed as required.

**Hazardous waste:** A solid waste that meets the definition of a hazardous waste under RCRA as defined in 40 CFR § 261.3.

**Investigation-derived waste (IDW):** Solid wastes, as defined in 40 CFR § 261.2, directly generated as result of performing the field activities.

**Nonhazardous waste:** A solid waste that does not meet the definition of a hazardous waste as defined in 40 CFR § 261.3 or is excluded from hazardous waste regulation per 40 CFR § 261.4(b).

**Soils and soil cuttings:** Solid material generated from excavation or drilling processes. Soils may include native soils, fill materials, and/or other historical plant waste streams used as fill materials on the site.

**Solid waste:** Any waste stream (solid, liquid or containerized gas) that meets the definition of solid waste under RCRA as defined in 40 CFR § 261.2.

#### **4.0 RESPONSIBILITIES**

This section presents a brief definition of the field team roles and responsibilities for management of IDW generated while conducting field investigations. This list is not intended to be a comprehensive list as additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Responsible to ensure that all field team members are properly trained per their responsibilities associated with IDW and that appropriate equipment and facilities are available for appropriate IDW management.

**Field Team Leader (FTL):** Implements the field program and supervises all field team members in the appropriate management of IDW. Ensures that only properly trained personnel are managing IDW on the site.

**Environmental, Health and Safety (EHS) Officer:** Assists the Team Leader in the supervision of all IDW management on site. The EHS officer shall be responsible for all IDW identification

and characterization, on site disposal, off site shipment and disposal, waste accumulation, emergency response and contingency planning, IDW training, and IDW reporting and recordkeeping.

**Project Team Members:** Ensure that they are properly trained prior to any IDW management as well as follow the appropriate IDW procedures and training.

## **5.0 REGULATORY BASIS AND GUIDANCE**

IDW encountered, generated, or managed during the field investigations may contain hazardous substances as defined by CERCLA. Some IDW may be hazardous wastes under RCRA while others may be regulated under other federal laws such as TSCA. These regulatory requirements may be applicable or relevant and appropriate requirements (ARARs) which impact how the IDW is managed.

### **5.1 EPA Guidance on IDW Management**

The management of IDW shall be in accordance with EPA Guidance “Management of Investigation-Derived Wastes During Site Inspections”, May 1991 (EPA, 1991). The specific elements of EPA’s guidance for IDW management are as follows:

- Characterizing IDW through the use of existing information (manifests, MSDSs, previous test results, knowledge of the waste generation process, and other relevant records) and best professional judgement.
- Delineating an AOC unit for leaving RCRA hazardous soil cuttings within the unit.
- Containerizing and disposing of RCRA hazardous groundwater, decontamination fluids, PPE, and disposable equipment at RCRA Subtitle C facilities.

- Leaving on-site RCRA nonhazardous soil cuttings, groundwater, and decontamination fluids preferably without containerization and testing.

In general, EPA does not recommend removal of wastes from sites, in particular, from those sites where IDW do not pose any immediate threat to human health or the environment. Actions taken during field investigations with respect to IDW that leave conditions essentially unchanged should not require a detailed analysis of ARARs or assurance that conditions at the site after field investigations will comply with the ARARs. At the same time, field personnel ensure that their handling of IDW does not create additional hazards at the site.

In brief, compliance with the NCP can generally be assured by:

- 1) Identifying contaminants, if any, present in the IDW based upon existing information and best professional judgement; testing is not required in most circumstances.
- 2) Determining ARARs and the extent to which it is practicable to comply with them.
- 3) Delineating an AOC unit based upon existing information and visual observation if soil cuttings are RCRA hazardous.
- 4) Burying RCRA hazardous soil cuttings within the AOC unit, so long as no increased hazard to human health and the environment will be created. Containerization and testing are not required.
- 5) Containerizing RCRA hazardous groundwater and other RCRA hazardous IDW such as PPE, disposable sampling equipment, and decontamination fluids for off-site disposal.

## **5.2 Hazardous Waste Regulation**

The RCRA hazardous waste regulations are clearly ARARs for hazardous IDW generated and managed during field investigations. However, with the application of EPA IDW guidance, RCRA requirements apply to management of IDW in the following manner:

- If RCRA hazardous IDW is stored or disposed off-site, then comply with all RCRA (and other ARAR) requirements.
- If RCRA hazardous IDW is stored on-site, then comply with RCRA (and other ARAR) requirements to the extent practicable.

For these field investigations, the following general guidance is expected to be practicable and therefore followed, recognizing that each situation will be evaluated against EPA IDW guidance (EPA, 1991) as well as RCRA hazardous waste requirements and other ARARs:

- IDW may be assumed not to be a “listed” hazardous waste under RCRA 40 CFR 261 Subpart D, unless available information about the site suggests otherwise.
- IDW characterization to determine if the IDW exhibits RCRA hazardous waste characteristics do not typically require testing if the characterization can be made by “applying knowledge of the hazardous characteristics in light of the materials or processes used” or by historical testing consistent with 40 CFR § 262.11(c).
- Compliance with the RCRA hazardous waste generator requirements of 40 CFR Part 262 for all RCRA hazardous IDW generated and/or managed (with exception of soil cuttings managed in accordance with the EPA IDW guidance). It is presumed that the RCRA hazardous IDW generated will fall within the large quantity generator (LQG) requirements.
- Land disposal does not occur (and thus the Land Disposal Restrictions [LDR] of 40 CFR Part 268 are not applicable) when IDW soil cutting wastes are:

- Moved, stored or left in place within a single AOC unit;
  - Capped in place;
  - Treated in situ (without moving the IDW to another AOC unit for treatment); or
  - Processed within the AOC unit to improve structural stability (without placing the IDW into another AOC unit for processing).
- Conversely, land disposal does occur (and the LDR of 40 CFR Part 268 are applicable) when IDW soil cutting wastes are:
  - Moved from one AOC unit to another AOC unit for disposal;
  - Moved outside an AOC unit for treatment or storage and returned to the same AOC unit for disposal;
  - Excavated from an AOC unit and placed in a container, tank, surface impoundment, etc. and then re-deposited back into the same AOC.

### 5.3 TSCA PCB Regulation

IDW containing PCBs at detectable levels may be generated, although the concentration of PCBs in any IDW generated is expected to be far below 50 ppm. However, IDW generated will be evaluated for PCBs and managed according to the following per the requirements of 40 CFR Part 761 Subpart D:

- Liquid IDW at concentrations greater than or equal to 50 ppm PCBs will be incinerated off-site at a TSCA-approved incinerator site.
- Nonliquid IDW at concentration greater than or equal to 50 ppm PCBs may be incinerated, treated by an equivalent TSCA-approved method, or disposed in a TSCA chemical landfill off-site.
- IDW at concentrations less than 50 ppm are generally not regulated under TSCA, and may be disposed in an acceptable Subtitle D facility.

## **6.0 DESCRIPTION OF ANTICIPATED IDW MANAGEMENT**

The following subsections provide a description of the anticipated IDW to be encountered, generated, and/or managed at the Libby Asbestos Superfund Site OU3 during field activities and the anticipated management of each. It should be noted that this information is provided for planning purposes, and will be evaluated and may need to be revised based upon actual experience while on site.

### **6.1 Soil and Soil Cuttings**

During field investigations, surface soil samples, samples of mine waste rock, and samples of fine tailings will be collected. Only a small portion of material will be collected for analysis. While the soil and soil cuttings IDW generated will be evaluated on a case-by-case basis, the general approach will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Characterizing the IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the waste generation process, and other relevant records) and best professional judgement.
- Soil and soil cuttings which are not used directly for sample makeup will not be taken outside of the AOC unit in which they were generated.
- Soil and soil cuttings within the AOC where they are generated will be placed back into the same investigation pit, trench, or bore hole and in the same order from which the material was removed, to the extent practicable and unless noted otherwise in the FSP.
- Soil cuttings potentially requiring RCRA disposal will be handled per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

### **6.2 Spent Sampling-Related Equipment**



During field investigations, spent sampling-related equipment will be generated. This may include (but not limited to) plastic sheeting/tarps, rope, bailers, sampling equipment, spent PPE, sample bottles, used containers, packaging materials, and other consumables. Although the vast majority of the spent sampling-related equipment is expected to be nonhazardous, these IDW may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the spent sampling-related equipment will be evaluated on a case-by-case basis, the general approach to be followed for spent sampling-related equipment IDW will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Containerizing the spent sampling-related equipment, typically in a satellite accumulation station.
- Characterizing the spent sampling-related equipment IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the contaminants present, and other relevant records) and best professional judgement. This characterization will be documented and maintained as part of the solid/hazardous waste determination records.
- Those spent sampling-related equipment IDW that are determined to be nonhazardous will be disposed of onsite or as municipal waste.
- Those spent sampling-related equipment IDW that are determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

### **6.3 Decontamination Fluids and Solids**

During field investigations, decontamination fluids and solids will be generated. Typically, these will be generated at a common decon area, although there may be more than one decon

area. Typically, the decontamination IDW will include (but not limited to) washwater from vehicles/equipment, and cleaning agents. Although the vast majority of decontamination IDW is expected to be nonhazardous, this IDW may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the decontamination IDW will be evaluated on a case-by-case basis, the general approach to be followed for decontamination IDW will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Containment of decontamination fluids (typically washwater) as generated. The washwater will be segregated from solids to the extent practicable (i.e., solids will be allowed to settle out of the washwater on the decontamination containment pad). Washwater will then be containerized to await waste determination. Solids will also be containerized in a separate container to await waste determination.
- Other decontamination solids such as cleaning utensils and PPE will also be containerized to await waste determination.
- Characterizing the decontamination IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the contaminants present, and other relevant records) and best professional judgement. This characterization will be documented and maintained as part of the solid/hazardous waste determination records.
- The decontamination solids IDW that are determined to be nonhazardous will be disposed of onsite.
- The decontamination liquids IDW that are determined to be nonhazardous will be disposed as a nonhazardous solid waste, preferably on-site.

- The decontamination IDW (either liquid or solid) that are determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

#### **6.4 Drilling, Well Purging, and Development Water**

Generally, water at the Site that is extracted from boreholes, wells or piezometers for the purpose of drilling, development, sampling, or hydraulic testing is considered non-hazardous and will be discharged to designated shallow sumps away from the boreholes or wells at the site. If the water generated is determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

### **7.0 PROCEDURES FOR HAZARDOUS IDW MANAGEMENT**

The following procedures apply to all IDW that have been determined to be hazardous except for soil cuttings IDW that remain with the AOC unit.

#### **7.1 Introduction**

Once an IDW has been determined to be hazardous, the federal RCRA Subtitle C waste management requirements apply to that waste. The scope of this procedure covers the requirements for large quantity generators (LQG) of hazardous IDW which manage the hazardous IDW on site such that RCRA permitting is not required.

#### **7.2 Determine Land Disposal Restrictions**

The 1984 amendments to the RCRA law included a prohibition of land disposal of certain hazardous wastes without first meeting some treatment standards. For the most part, all listed and characteristic hazardous wastes must be treated according to the treatment levels and technologies outlined in 40 CFR Part 268 to reduce the toxicity and/or mobility of hazardous constituents prior to being disposed of on the land, i.e., landfilled. Therefore, a generator must

determine if the waste is a "restricted waste" under the land ban rules, and if so, off site treatment and disposal is limited. Note that these rules apply only to wastes destined for land disposal which is defined as: placement in or on the land including a landfill, surface impoundment, waste pile, injection well, land treatment facility, salt dome formation, salt bed formation, underground mine or cave, or concrete vault or bunker. Wastes which are shipped off site for disposal other than land disposal are not regulated under the land disposal restriction regulations of 40 CFR Part 268.

Generators of hazardous wastes must determine if the waste is restricted from land disposal under 40 CFR Part 268. The following reporting and recordkeeping requirements apply.

- If a generator determines that he is managing a restricted waste and the waste does not meet the applicable treatment standards, with each shipment of waste, the generator must notify the treatment or storage facility in writing of the appropriate treatment standards;
- If the generator determines that he is managing a restricted waste and the waste can be disposed without further treatment, with each shipment of waste, the generator must submit to the treatment, storage or disposal facility a notice and certification stating that the waste meets the applicable treatment standards;
- If the generator determines that he is managing a waste subject to an exemption from a prohibition on the type of land disposal method utilized for the waste, with each shipment of waste, the generator must submit to the receiving facility a notice stating that the waste is not prohibited from land disposal;
- If the generator is managing prohibited waste in tanks, containers, or containment buildings regulated under 40 CFR 262.34, and is treating such waste in such tanks, containers, or containment buildings to meet applicable treatment standards, the generator must develop a waste analysis plan which describes the procedures the generator will carry out to comply with the treatment standards; and

- If the generator determines whether the waste is restricted based solely on his knowledge of the waste, all supporting data used to make this determination must be retained on-site in the generator's files.

The generator must retain on-site a copy of all notices, certifications, demonstrations, waste analysis data, and other documentation produced pursuant to these requirements for at least three years from the date the waste was last shipped from the site. It should also be noted that it is prohibited to dilute a hazardous waste in order to circumvent the land disposal prohibitions (40 CFR 268.3). Once a waste is determined to be a "restricted waste", an appropriate Treatment, Storage, and Disposal Facility (TSDF) can be selected to properly treat and dispose of the waste.

### **7.3 On-Site Accumulation**

As discussed in Section 5.0 above for each IDW generated, a large quantity generator (LQG) must make the appropriate hazardous waste determination per 40 CFR Part 262.11. If the IDW is determined to be hazardous, then the IDW will typically be stored on-site prior to shipment off-site for disposal. The following requirements apply to all hazardous IDW being stored on-site prior to shipment.

#### **7.3.1 EPA Identification Number (40 CFR Part 262.12)**

Any facility which is a LQG of hazardous wastes must not treat, store, dispose, transport or offer for transportation any hazardous waste without first obtaining a EPA identification number from EPA (or the authorized state). Hazardous wastes cannot be offered to transporters or to treatment, storage or disposal facilities that have not received a EPA identification number.

#### **7.3.2 On-Site Hazardous Waste Accumulation (Storage) (40 CFR 262.34(d))**

Two types of accumulation areas for hazardous waste are permissible for a LQG without RCRA interim status or a Part B permit. These are the "90-day storage area" and the "satellite accumulation station" (SAS). The SAS requirements are discussed below. With regards to a

"90-day storage area", a LQG may store hazardous wastes on-site for up to 90 days or less in a storage area, provided that the following conditions are met:

- If the waste is placed in containers, the requirements of 40 CFR Part 265 Subpart I (container requirements) are met. See below for container requirements;
- If the waste is placed in tanks, the requirements of 40 CFR 265 Subpart J (tank requirements) are met. See below for the tank requirements.
- At closure, the generator closes the storage area per the requirements of 40 CFR 265.111 and 40 CFR 265.114;
- The date which the hazardous waste is placed in the storage area is clearly marked on the container, and the container is clearly marked as "Hazardous Waste";
- The facility complies with 40 CFR Part 265 Subpart C, Preparedness and Prevention (See Section 6.3.3 below);
- The facility complies with 40 CFR Part 265 Subpart D, Contingency Plan and Emergency Procedures (See Section 6.3.4);
- The facility complies with 40 CFR Part 265.16 training requirements (See Section 6.6 below);
- Any hazardous wastes which are stored longer than 90 days must first be granted an extension by EPA (or authorized state).

#### **90-Day Storage Area Container Requirements (40 CFR Part 265 Subpart I)**

Hazardous waste stored in containers must meet the following requirements:

- Containers must be in good condition, free of leaks;
- Hazardous wastes must be compatible with container (or liner) material;
- Containers must always be kept closed except to add or remove wastes;

- Containers must be handled in a manner to avoid ruptures;
- The storage area must be inspected at least weekly to check for container deterioration; and
- Incompatible wastes must be stored separately with separate secondary containment.

Incompatible wastes are wastes that are unsuitable for co-mingling because the co-mingling could result in any of the following:

- Extreme heat or pressure generation;
- Fire;
- Explosion or violent reaction;
- Formation of substances that have the potential to react violently;
- Formation of toxic dusts, mists, fumes, gases, or other chemicals; and/or
- Volatization of ignitable or toxic chemicals due to heat generation.

#### **90-Day Storage Area Tank Requirements (40 CFR Subpart J)**

LQGs that accumulate or store hazardous wastes in tanks or tank systems must meet the following requirements:

- For tanks existing prior to July 14, 1986, an assessment of tank must be performed and certified by an independent, qualified, licensed engineer. The written certification must be kept on file at the facility (40 CFR 265.191);
- New tank systems (those built after July 14, 1986) must meet tank technical standards and have been certified by an independent, qualified, licensed engineer. The written certification must be kept on file at the facility (40 CFR 265.192);

- New tank systems must have adequate secondary containment and leak detection systems. Existing tanks must be upgraded to meet these standards by the time the tank is 15 years of age (40 CFR 265.193);
- Tanks must be operated to prevent system failure, overflow and spills. Tanks must be operated with sufficient freeboard to prevent overtopping (40 CFR 265.194);
- Inspect the tanks at least once each operating day for the following:
  - Discharge control equipment;
  - Monitoring equipment and controls;
  - Tank level; and
  - Evidence of leaks or spills. (40 CFR 265.195)
- Inspect the tanks at least weekly for corrosion, erosion or leaks;
  - The tank must meet the closure and post-closure care provisions of 40 CFR 265.197; and
  - Store incompatible wastes separately (40 CFR 265.199).

#### **Satellite Accumulation Station (SAS) Requirements (40 CFR 262.34(c))**

A SAS is a container placed at or near the point of waste generation for the purpose of collecting the waste as it is being generated. For example, a container may be placed in the quality control laboratory for collection of hazardous wastes generated in the laboratory. This SAS may collect up to 55 gallons of hazardous waste or 1 quart of acute hazardous waste. The SAS does not need to meet the requirements of a storage area, provided the following conditions are met:

- The amount of hazardous waste accumulated at the SAS does not exceed 55 gallons (or 1 quart of acute hazardous waste);



- The SAS is located at or near the point of generation where the waste is initially accumulated and is under the control of the operator of the process generating the waste;
- The container used is in good condition, is compatible with the wastes being accumulated, and is kept closed except to add or remove wastes;
- The container is marked with the words "Hazardous Waste" or other words to identify the contents; and
- Once the 55-gallon limit is reached, the date is marked on the container and the container is moved from the SAS within three days to a proper location. For example, the wastes must either be moved to the storage area or be picked up by a waste transporter and moved off-site.

### **7.3.3 Preparedness and Prevention (40 CFR Part 265 Subpart C)**

The following preparedness and prevention steps must be taken concerning the hazardous waste storage area:

- The storage area must be operated and maintained to minimize the possibility of fire, explosions or releases of hazardous waste;
- The facility must have appropriate communication systems, fire-fighting equipment, spill control equipment and decontamination equipment;
- All emergency response systems and equipment must be tested monthly with documentation and maintained to assure proper operation;
- Persons handling hazardous wastes must have immediate access to alarms and/or communication systems;
- The storage area shall have adequate aisle space for emergency response activities; and

- The facility must attempt to make arrangements with the local police, fire departments, emergency response teams, and local hospitals to assure readiness for potential emergencies associated with the storage area.

#### **7.3.4 Contingency Plan and Emergency Procedures (40 CFR Subpart D)**

A LQG that accumulates or stores hazardous waste on site in a 90-day storage area must develop and keep current a contingency plan for the facility. The purpose of the contingency plan is to provide an organized plan of action and delegation of responsibilities and authority to specific facility personnel to respond to emergency situations that may require both the facility and/or outside resources. The contingency plan is designed to minimize hazards to humans or the environment from fires, explosion or any unplanned sudden or non-sudden release of hazardous waste/hazardous waste constituent to air, soil or surface water in compliance with the requirements of 40 CFR 265 Subpart D. A Contingency Plan will be maintained on the site if hazardous IDW are accumulated on-site.

The key components of the contingency plan include the following (40 CFR 265.52):

- A description of the emergency response organization, including designation of the Emergency Coordinator and alternates;
- Response procedures;
- Emergency notification;
- Arrangements with local authorities;
- List of names, addresses and phone numbers of designated emergency personnel and alternates;
- List of emergency response communication equipment and locations;
- Evacuation procedures, routes and alternates; and
- Procedures for amending the plan.

Copies of the plan must be sent to (40 CFR 265.53):

- The Project Manager;
- Lincoln County Sheriff's department;
- Libby fire department; and
- Other agencies as deemed appropriate.

The emergency coordinator (EC) is the key person facilitating emergency preparedness and response. The EC or designated alternate shall be on-site or on-call at all times. The EC and alternates must be trained and thoroughly familiar with the contingency plan, emergency response activities and operation of the facility. The EC must know the locations and characteristics of all waste generated, location of all records within the facility and the facility layout. The EC must have the authority to commit the resources needed to carry out the spill response plan. Any person or department who first discovers any spill of a hazardous waste/material is responsible for notifying the spill response/emergency response coordinator. The EC for the Libby Mine Site field investigations will be the EHS Officer with the Field Team Leader and the Project Manager as alternates.

The contingency plan should be reviewed and immediately amended when:

- Changes in applicable regulations occur;
- The plan fails in an emergency;
- Changes are made to emergency procedures;
- Changes occur in emergency personnel list; or
- Changes occur in emergency equipment list.

#### **7.4 Pre-Transportation Requirements**

Prior to transporting hazardous wastes or offering hazardous wastes for transportation off-site, the generator must comply with the following:

- Package the hazardous wastes in DOT-approved containers per 49 CFR Parts 173, 178 and 179. DOT-approved containers (such as drums) are usually marked as being DOT-approved);
- Label the hazardous wastes according to DOT labeling requirements per 49 CFR Part 172;
- Mark each container (of 110 gallons or less) used in transportation with the following:

HAZARDOUS WASTE - Federal Law Prohibits Improper Disposal. If found, contact the nearest police or public safety authority or the EPA.

- Generator's Name and Address
- Manifest Document Number
- Ensure that the initial transporter placards the transport vehicle with the appropriate placard in accordance with 49 CFR Part 172 Subpart F.

### **7.5 Manifesting Off-Site Shipments of Hazardous IDW**

Any generator which transports or offers for transportation hazardous waste for off-site treatment, storage or disposal must prepare a manifest according to manifest instructions for each shipment of similar hazardous wastes. The manifest must be carefully filled out with each shipment. Take care to follow the instructions and use the terms as listed in the instructions. A generator must designate on the manifest one facility (designated facility) which is permitted to handle the waste described on the manifest (40 CFR 262.20).

The generator must determine if the state to which the wastes are destined (consignment state) requires use of its own manifest. If so, then the consignment state's manifest must be used. If the consignment state does not require use of its manifest, and the state in which the waste

shipment originates (generator state) does, then the manifest from the generator state must be used. If both states have manifests, use the consignment state manifest, making sure that there are sufficient copies to meet the generator state distribution requirements. If neither state requires use of its manifest, then any uniform hazardous waste manifest may be used (40 CFR 262.21).

The manifest must contain at least enough copies such that the generator gets two copies, the transporter gets one copy and the designated facility gets one copy. Some states require additional copies to be sent to the state. At the time of shipment, the generator must keep one copy (the generator copy) of the completed, signed manifest and give the remaining copies to the transporter. Each copy must have the signature of the generator and the transporter at the time of shipment. The original manifest shall be returned to the generator once the shipment reaches the designated facility and the manifest is signed by the designated facility (40 CFR 262.21).

If the original, signed manifest is not received by the generator within a certain number of days, action by the generator is required. These requirements are discussed in the following sections:

- If, after 35 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must contact the transporter and/or the designated disposal facility to determine the status of the hazardous waste (40 CFR 262.42(a)(1)).
- If after 45 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must submit an exception report to the U.S. EPA (or authorized state). The exception report must include a copy of the manifest along with an explanation of efforts to locate the hazardous wastes and the result of these efforts (40 CFR 262.42(a)(2)).

## **7.6 Personnel Training**

Any person, and their immediate supervisor(s), involved in waste management at a LQG facility which stores hazardous waste in a 90-day storage area must undergo initial and annual training

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for hazardous waste management (40 CFR 262.34(a)(4) and 40 CFR 265.16). Facility personnel are required to successfully complete a program of classroom instruction or on-the-job training that teaches them to perform hazardous waste management duties relevant to their jobs. The program must be directed by a person trained in hazardous waste management procedures.

The training must be designed to enable personnel to effectively respond to emergencies by becoming familiar with emergency procedures, emergency equipment and emergency systems, including the following;

- Procedures for using, inspecting, repairing and replacing facility emergency and monitoring equipment;
- Communications or alarm systems;
- Response to fires or explosions; and
- Off-site communication.

Employee training is to be held at regular intervals. Emergency planning information, e.g., the Contingency Plan, also should be provided to state and local emergency response agencies at regular intervals (40 CFR 265.37 and 265.53). Employees required to receive the training cannot work unsupervised until they have completed the training requirements (either classroom or on-the-job training). In addition, facility personnel must take part in an annual review of the initial training.

The following records must be maintained at the facility for employees affected by this training:

- Job title for each position and name of employee filling each job;
- Job descriptions for each position related to hazardous waste management;
- Written description of type and amount of initial and continuing training that will be given to each person filling the various job positions; and

- Documentation that necessary training has been given and completed by each affected personnel.

Training records are required to be kept on current personnel until closure of the facility. For former employees, training records must be kept for at least three years from the date the employee last worked at the facility and may be transferred if the employee stays within the same company (40 CFR 265.16(e)).

## **7.7 Reporting and Recordkeeping**

The following reports are required of a LQG:

- Manifest exception reports as discussed in Section 6.5 above.
- A LQG must submit a Biennial Report to the EPA (or authorized state) every even numbered year by March 1, e.g., March 1, 2008 for the 2007 reporting year. The Biennial Report is to be submitted on EPA form 8700-13A.

The following records are required to be kept for a minimum of three years by the LQG:

- The signed original manifests;
- Biennial reports;
- Exception reports;
- All records pertaining to hazardous waste determinations; and
- Land disposal determination records, notification and certification records.

## **8.0 QUALITY ASSURANCE AND QUALITY CONTROL**

All IDW data must be documented in the field logbooks, field forms, manifests, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will

check and verify that IDW documentation has been completed per this procedure and other procedures referenced herein.

## **9.0 REFERENCES**

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

EPA, 1991. Management of Investigation-Derived Wastes During Site Inspections, EPA May 1991, EPA/540/G-91/009



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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: May 29, 2008

OU3 SOP 14 (Rev. 0)

Title: INSTALLATION, OPERATION, AND MAINTENANCE OF THE AUTOMATED  
WATER SAMPLING AND FLOW MONITORING DEVICES

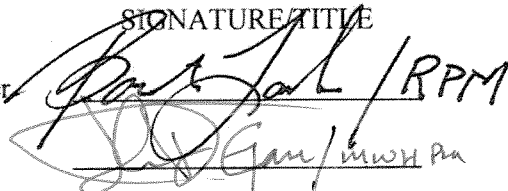
**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager

 /RPM

6/3/08

SOP Author

 /mwh pm

6-04-08

Revision Number	Date	Reason for Revision
0	05/29/2008	--

## 1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the protocols to be followed when installing, operating, and/or maintaining an automated water sampling device including a flow monitoring device used for collecting flow data and flow based water samples.

This document focuses on methods and equipment that are readily available and typically applied for the installation, operation, and maintenance of automated water sampling and flow monitoring devices. It is not intended to provide an all-inclusive discussion regarding automated water sampling and flow monitoring devices. Specific installations and operational problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project specific sampling plan and approved by the Project Manager and the Quality Manager.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in installation, operations, and/or maintenance of automated water sampling and flow monitoring devices must adhere to health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## 3.0 DEFINITIONS

**Automated Water Sampling Device:** Isco 6712 Full-size Portable Sampler ([Attachment 1](#)). A stand alone electronic device that when coupled with a flow monitoring device is capable of collecting flow or time based water samples. It is capable of collecting various quantities of water over various durations of time and is capable of recording data over time via external instruments (in this case a flow monitoring device).

**Flow Monitoring Device:** Isco 720 Submerged Probe Flow Module ([Attachment 2](#)). An electronic device that uses a pressure transducer to measure the level of the flow stream then converts the depth measurement into flow rate.

## 4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FS), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Selects the specific sampling location (within the specified sampling area) for the automated sampling device.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the installation of the automated water sampling and flow monitoring device, performs the calibration of the automated water sampling and flow monitoring device, and ensures that data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer:** Provides oversight for the automated sampling and flow monitoring device installation, supervises other personnel, and ensures compliance with SOPs and QA/QC requirements. The FTL prepares daily logs of field activities.

**Sampling Technician (or other designated personnel):** Assists the FTL in the implementation of tasks. The Sampling Technician performs the actual automated water sampling and flow monitoring device installation, maintenance, and documentation.

## **5.0 AUTOMATED WATER SAMPLING AND FLOW MONITORING DEVICE INSTALLATION, OPERATION, AND MAINTENANCE PROCEDURES**

This section describes typical automated water sampling and flow monitoring device installation, operation, and maintenance procedures.

### **5.1 Installation**

Installation of an automated water sampling and flow monitoring device includes the following 5 steps: (1) identify an appropriate location for the automated sampling device, (2) install the automated sampling device, (3) mount the flow monitoring device in the stilling well attached to the flume, (4) calibrate the automated sampling device, (5) Program the automated water sampling device. These steps are described within this Section.

#### **5.1.1 Identify a Location**

An ideal location for the automated sampling device is a flat surface above the high flow line of the channel, no more than twenty-five feet from the flume. The area should be large enough to allow the Sampling Technician to safely access the device for operation and maintenance. An ideal location for the automated sampling device is directly adjacent to the flume with no curves in the suction line (Figure 1a, Figure 1b).

The actual location will be identified by the Project Manager and other key personnel who know the objectives of the flow based water collection and flow monitoring. The actual location may not be the ideal location due to the objectives. If it is necessary to curve the suction line, curve angles will be no greater than forty-five degrees.

#### **5.1.2 Install the Automated Water Sampling Device**

The manufacturer's instructions should be followed during installation. This section contains an overview of a typical installation.

The first step is installing the distributor arm and discharge tube. The distributor arm location is dependant on the sample container configuration.

The second step is configuring the automated sampler for the appropriate sample container. For this application a single bottle configuration will be used for all flow based sampling. Samples will be collected in single-use 2 gallon sample bags.

The third step is installing the power source and when appropriate (sampling location LRC-06 only) the solar panel/battery charger. The battery voltage will be recorded when the battery is installed and the voltage will be recorded when the solar panel/battery charger is installed.

The fourth step is installing the suction line. The suction line will be attached to the pump tube on the automated water sampling device and attached to the flume. The suction line will be attached to the flume in the main current of the flow not in an eddy and not on the edge of the flow and not on the bottom of the channel. The suction line will be attached to the flume cross bar above the flow with zipties. The suction line will descend into the center of the flume. The intake of the suction line will be facing downstream. The automated water sampling device is capable of sampling water 1/4 inch in depth or deep enough to submerge the suction line. The suction line will be ideally routed in a straight line on a continuous downhill slope from the automated sampling device.

The fifth step is installing the instrument enclosure (sampling location LRC-06 only).

### **5.1.3 Mount the Flow Monitoring Device**

The manufacturer's instructions should be followed during installation. This section contains an overview of a typical installation.

The first step is connecting the flow monitoring device to the automated water sampling device. The Isco 6712 Full-sized Portable Sampler (automated water sampling device) and the Isco 720 Submerged Probe Flow Module (flow monitoring device) were designed to interact and the connection requires no converter or adjustments to either device.

The second step is determining the flow monitoring device mounting location on the flume. The flow monitoring device will be mounted on the stilling well wall, completely submerged, and immobile to ensure accuracy.

The third step is connecting the flow monitoring device cable to the automated water sampling device. Ideally the cable will run parallel to the suction line and both will be protected within a 4" PVC pipe. If the location of the automated water sampling device will not allow for the suction line and flow monitoring device cable to run in a straight line, the FTL will design an alternative protective device.

#### **5.1.4 Calibrate the Automated Water Sampling Device**

The manufacturer's instructions should be followed during calibration. This section contains an overview of a typical calibration.

The first step is configuring the automated water sampling device. Various information such as time and date, site name, length of suction line, type of flow monitoring device, etc. will be programmed in the automated water sampling device.

The second step is calibrating the sample volume. The Isco 6712 Full-size Portable Sampler observes the volume of water being collected by the number of rotations the pump completes. Because of this, the automated water sampling device must be calibrated to collect the desired volume of water. Calibrate the automated water sampling device by setting a sample volume and collecting it in a graduated cylinder. If the desired sample volume and the actual sample volume differ, make the necessary adjustments on the automated water sampling device.

The third step is configuring the flow monitoring device. Various information such as desired flow units, data storage time interval, the depth of water in the stilling well, the flume type, etc. will be programmed in the automated water sampling device.

The fourth step is calibrating the flow monitoring device. The flow is calculated using the staff gauge on the flume and the manufacturer's gauge-flow chart, compared to the flow observed on the automated water sampling device, and adjusted accordingly.

#### **5.1.5 Programming the Automated Water Sampling Device**

The manufacturer's instructions should be followed during calibration. This section contains an overview of typical programming.

The first programming step is to create a program defining the conditions that activate the automated water sampling device. Various information such as flow, number of bottles, sample volume, suction line length, etc. will be required to create the program.

The second programming step is to run a test sample. A program that will collect a sample and multiple flow readings in a short period of time will be run. The sample will be collected in a graduated cylinder and the recorded flow readings will be downloaded. The actual volume of the sample will be compared to the desired volume to verify the correct volume of water is being collected. The flow readings will be reviewed to verify the correct number of readings and that the values of the readings are the correct.

## **5.2 Operation**

Operating the automated water sampling and flow monitoring device consists of mainly routine data collection, and when necessary, sample retrieval. During data collection the accuracy of the flow monitoring device will be checked by recording the flow on the staff gauge and comparing it to the reading on the automated water sampling device. If the automated water sampling device has collected a sample, the sample will be removed, and the single-use container will be replaced. The sample will be distributed among the analytical bottle set as described in SOP No. 3-Surface Water Sampling.

## **5.3 Maintenance**

Maintenance of the automated water sampling and flow monitoring device includes:

- Check the stilling well for debris, sediment, or anything that may be affecting the flow monitoring device. This will be completed during each visit. If any of the above are identified, the problem will be resolved by removing the flow monitoring device and cleaning the stilling well and port.
- Check the suction line for kinks in the line or any damage. This will be completed during each visit. If any of the above are identified, the suction line will be replaced.



- Check the flow monitoring device cable for any damage. This will be completed during each visit. If any of the above are identified, the problem will be resolved by repairing or replacing the cable.
- Check the status of the desiccant chamber connected to the vented cable of the flow monitoring device. If the desiccant has been consumed (evident by color change), it will be replaced with fresh desiccant.
- Check the battery voltage and when necessary replace the battery. This will be completed during each visit.
- Check the solar panel and cable for any damage (sampling location LRC-06 only). This will be completed during each visit. If any of the above are identified, the solar panel and/or cable will be repaired or replaced.
- Check the flow and verify the accuracy of the flow monitoring device by comparing it to the staff gauge reading. This task will be completed only after the above tasks have been completed. This will be completed during each visit. If the flow is inaccurate, the automated water sampling device will be calibrated as described in Section 5.1.4.
- Check if the automated water sampling device program is correct and functioning properly. This will be completed during each visit. If the program is incorrect, the correct program will be selected. If the program is not functioning properly, the program will be recreated.
- Check the sample container for any damage and that it is properly attached to the automated water sampling device. This will be completed during each visit. If the sample container is damaged, it will be replaced. If the sampling device is incorrectly attached the container will be visually observed for possible contamination and either re-attached or replaced.
- Check the discharge tube for any obstructions or damage. This will be completed during each visit. If any of the above are identified, the discharge tube will be replaced.

All maintenance activities must be recorded in the field logbook or on field forms following SOP No. 9-Field Documentation.

## **6.0 QUALITY ASSURANCE AND QUALITY CONTROL**

The Isco 720 Submerged Probe Flow Module (flow monitoring device) is accurate to  $\pm 0.002$  feet. The accuracy of the flow monitoring device will be affected by debris, sediment, and any obstruction in the stilling well or contacting the flow monitoring device. Maintenance activities must be evaluated to determine if the problems were severe enough to qualify the records obtained. Maintenance activities must be directed toward determining if the problems were associated with the flow monitoring device or the automated water sampling device. This is important because maintenance and the observation of the problems will have taken place following the recording of the data. The cause of the problem must be determined prior to another data collection period.

## **7.0 DECONTAMINATION**

All equipment used in the installation or sampling process shall be decontaminated prior to field use and after field use. Decontamination procedures are presented in SOP No. 7-Equipment Decontamination. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated shall be managed in accordance with the procedures outlined in SOP No. 12- Investigative Derived Waste Management.

## **8.0 REFERENCES**

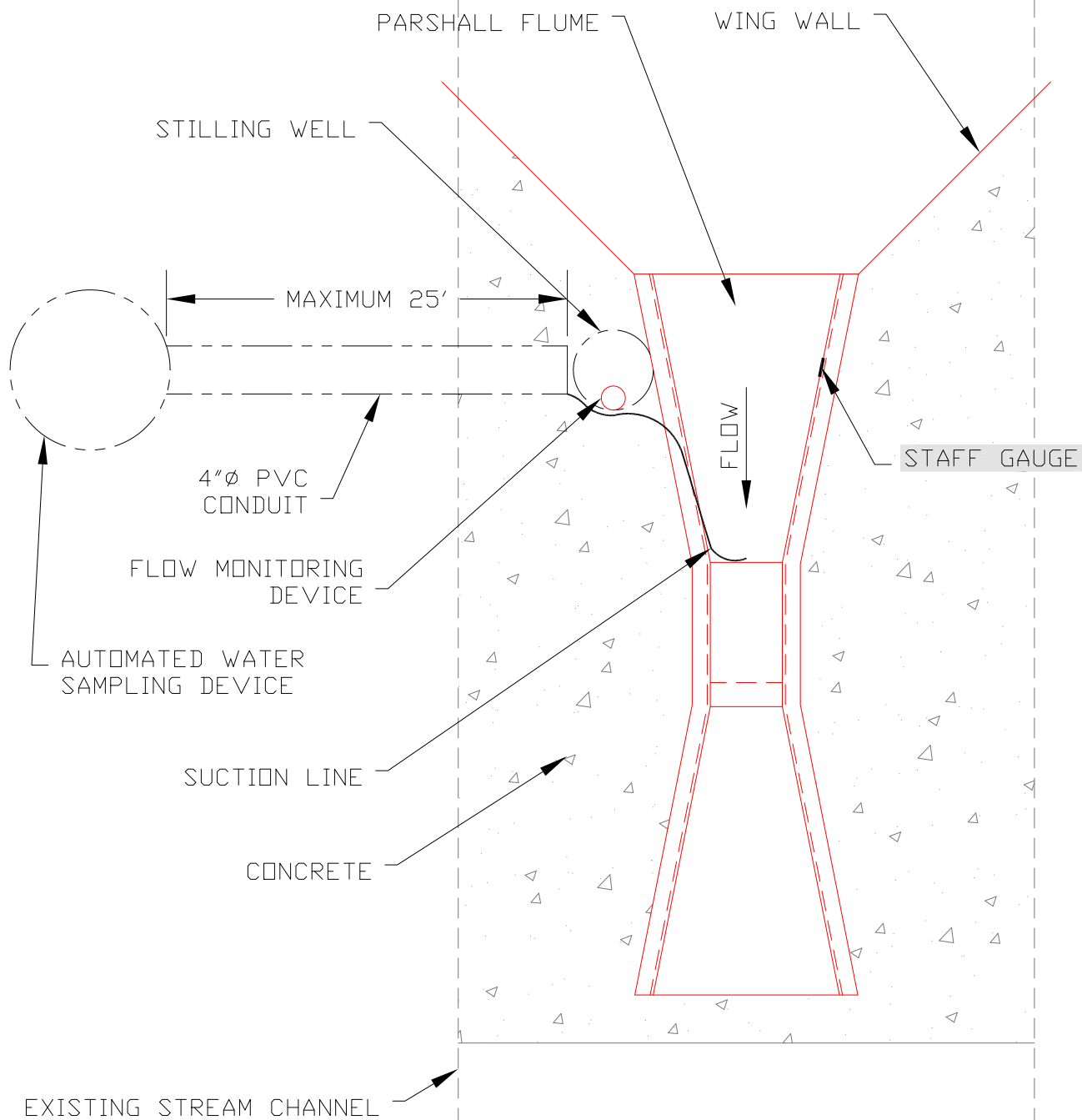
Agency for Toxic Substances and Disease Registry (ATSDR). 2006. Asbestos Exposure and Your Health.


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**FIGURE 1A**

**AUTOMATED WATER SAMPLING AND FLOW MONITORING DEVICE  
INSTALLATION DETAILS**

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REV. No.	REVISIONS	REV. DATE	DESIGN BY	DRAWN BY	REVIEWED AND SIGNED BY
 <b>MWH</b>			PROJECT No.:		
			AutoCAD FILE:		
			SCALE: N.T.S.		
			FIGURE: 1a		

LIBBY ASBESTOS SUPERFUND SITE  
DU3 PHASE IIA REMEDIAL INVESTIGATION

AUTOMATED WATER SAMPLING AND  
FLOW MONITORING DEVICE INSTALLATION DETAILS

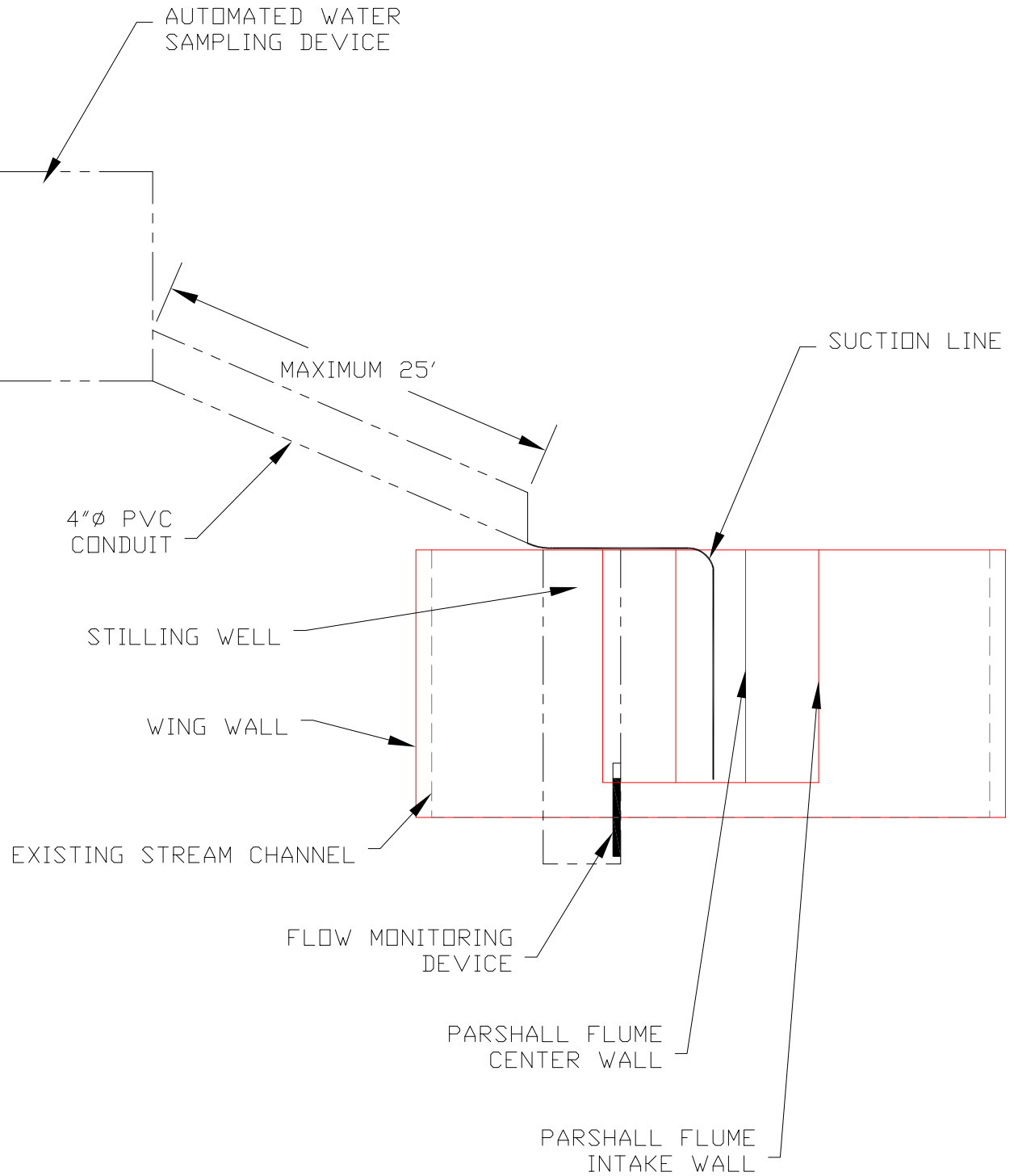
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**FIGURE 1B**

**AUTOMATED WATER SAMPLING AND FLOW MONITORING DEVICE  
INSTALLATION DETAILS**



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REV. No.	REVISIONS	REV. DATE	DESIGN BY	DRAWN BY	REVIEWED AND SIGNED BY
 <b>MWH</b>		PROJECT No.:			
		AutoCAD FILE:			
		SCALE: N.T.S.		FIGURE: 1b	

LIBBY ASBESTOS SUPERFUND SITE  
DU3 PHASE IIA REMEDIAL INVESTIGATION

AUTOMATED WATER SAMPLING AND  
FLOW MONITORING DEVICE INSTALLATION DETAILS

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**ATTACHMENT 1**

**ISCO 6712 FULL-SIZE PORTABLE SAMPLER**

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# Isco 6712 Full-size Portable Sampler

Isco's 6700 Series Portable Samplers have set the industry standard, providing the most comprehensive and durable performance available. With the introduction of our new 6712, Isco takes another step toward the ultimate by including SDI-12 interface capabilities.

The 6712 uses Isco's advanced 6700 Series Controller, a device that allows you to select from a variety of programming modes, assuring the most suitable routine for your application. Programming is fast and simple, with on-line help just a key stroke away.

The environmentally-sealed 6712 controller delivers maximum accuracy and easily handles all of your sampling applications, including:

- wastewater effluent
- stormwater monitoring
- CSO monitoring
- permit compliance
- pretreatment compliance

In the Standard Programming Mode, the controller walks you through the sampling sequence step-by-step, allowing you to choose all parameters specific to your application. Selecting the Extended Programming Mode lets you enter more detailed programs.

An optional telephone modem allows programming changes and data collection to be performed remotely, from a touch-tone phone. It also has dial-out alarm features.

*Bottle options are available for practically any sequential or composite application.*



## *Versatile and Convenient*

With eleven bottle choices, Isco's 6712 Sampler lets you quickly adapt for simple or intricate sampling routines. Up to 30 pounds (13.5 kg) of ice fits in the insulated base, preserving samples for extended periods, even in extreme conditions. A convenient drain plug aids removal of water from melted ice.

## *Tough and Reliable*

The 6712 Portable Sampler features a vacuum-formed ABS plastic shell to withstand exposure and abuse. Its tapered design and trim 20-inch (50.8 cm) diameter result in easy manhole installation and removal. Large, comfortable handles make transporting safe and convenient—even when wearing gloves.

Isco's 6712 Portable Sampler carries a NEMA 4X, 6 (IP67) enclosure rating. It's submersible, watertight, dust-tight, and resistant to sleet and corrosion.

Superior capability, rugged construction, and unmatched reliability make the 6712 the ideal choice for portable sampling in just about any application.

## All 6712 Samplers share the following features:

### ***Advanced Delivery System***

The 6712's peristaltic pump delivers samples at the EPA-recommended velocity of 2 ft/sec., even at head heights of 26 feet. At a head height of 3 feet, line velocity is 3 ft/sec. No other automatic sampler achieves this level of performance!

Our patented\* pump revolution counter tells you when tubing should be replaced. Changing tubing is a snap; there are no pump covers, collars or tools to slow you down. An exclusive safety interlock removes power from the pump when it's opened.

### ***Step-by-Step Programming***

This feature walks you through the sampling sequence and allows you to choose all parameters specific to your application:

- When to start
- What volume to collect
- How to distribute samples
- If samples are to be time- or flow-paced.

You can easily enter complex programs to suit your unique needs. Available routines include:

- Pause and resume for intermittent discharge flow monitoring
- Sampler pacing by time, non-uniform time, flow or external event
- Random interval sample collection

### ***Convenient Data Retrieval***

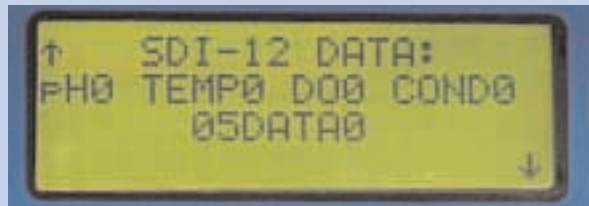
Every 6712 Sampler is also a powerful data logger. Sampling, flow, rainfall, and other water quality data can be stored in its 512 KB memory.

Data may be retrieved directly into a Flowlink® 4 equipped PC in three ways:

- Via cable connection
- Remotely, via Isco's 2102 Wireless Communication System
- By phone, using our optional built-in modem

### ***SDI-12 Interfacing***

The 6712 functions as a SDI-12 logger and connects to any sensor that fully implements the protocol standard.



*Display window showing SDI-12 connection status.*

In addition, Isco has defined extended commands to enable "plug and play" communications and ease of programming. These commands are implemented by the sensor manufacturer. Data are identified and logged by their specific type.

## Expand your monitoring capabilities with these products and accessories.

Contact Isco or your Isco Representative to receive specific literature and prices on the following items.

### ***Telephone Modem***

A factory-installed option that lets you set up and make programming changes, or collect data from your 6712 sampler from the comfort of your office.

### ***581 RTD (Rapid Transfer Device)***

Slim enough to fit in your shirt pocket, yet rugged enough to withstand submersion, the 581 RTD lets you quickly retrieve and transfer data without taking your laptop computer into the field.



### ***ProPak™ Disposable Sample Bags***

Isco's patented ProPak bags eliminate the expense of washing and storing bottles, while taking away worries about contamination from previous samples. The bags are available with a 1000 ml capacity, or in a 2-gallon version for composite sampling.

### ***Flowlink Software***

Isco's advanced Flowlink® 4 for Windows Data Management Software harnesses the power of Microsoft Windows® to retrieve, import, compare, and analyze data, generate advanced charts and graphs, create comprehensive reports, and more.

## 700 Series Modules

Our interchangeable 700 Series Modules let you adapt your 6712 sampler for a variety of jobs. These compact modules are environmentally sealed and may be added to your 6712 system at any time.



### 701 – pH and Temperature Module

Combines accurate pH and temperature monitoring in one module. It will also activate your 6712 Sampler at a user-elected pH or temperature range.

### 710 – Ultrasonic Flow Module

Uses our field-proven ultrasonic level sensor that doesn't require submersion in the flow stream.

### 720 – Submerged Probe Flow Module

Provides accurate measurement at sites where wind, steam, foam, turbulence, or air temperature fluctuations exist. Suitable for small channels, it accurately senses pressure even when covered with silt and sand.

### 730 – Bubbler Flow Module

Get the dependability and accuracy of Isco bubbler flow meters in a miniaturized package. The 730 is unaffected by changing stream conditions, and level measurement remains accurate despite temperature fluctuations or exposure to harsh chemicals.

### 750 – Area Velocity Flow Module

Gives greater accuracy where weirs and flumes are not practical, and where submerged, full pipe, surcharged, and reverse flow conditions may occur. And, you don't have to estimate the slope and roughness of the channel.

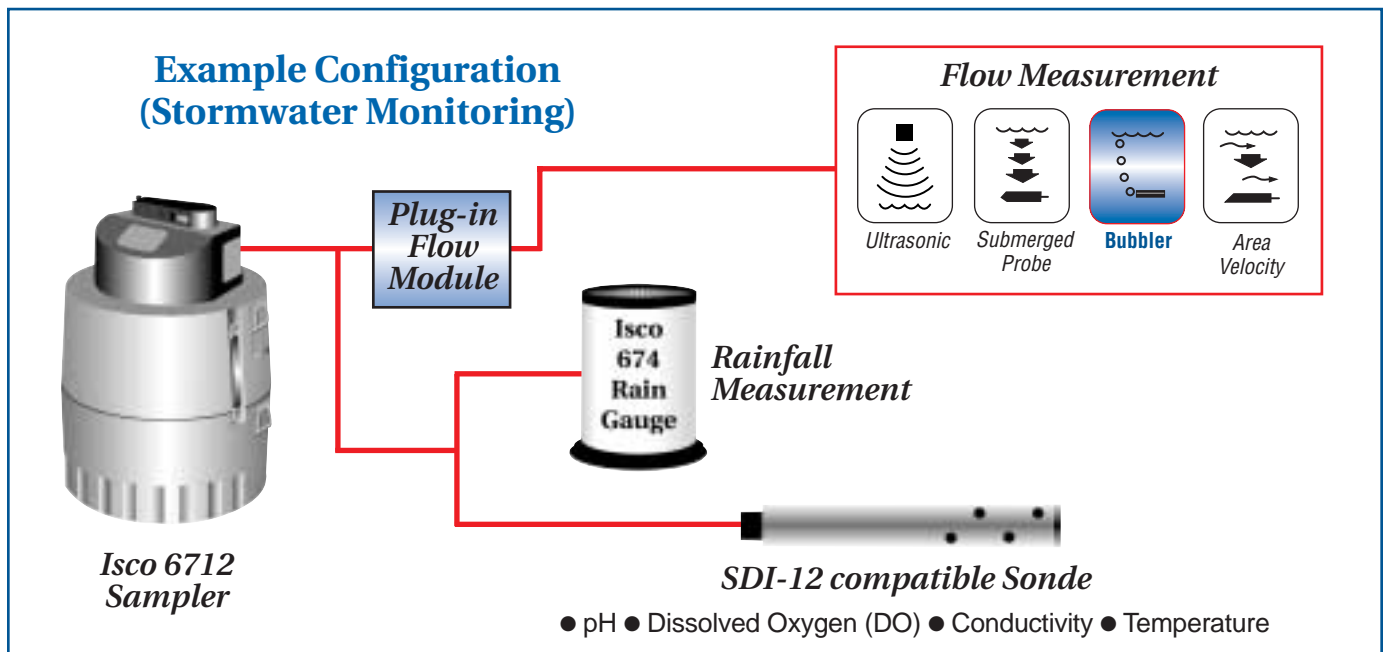
### 780 – Smart 4-20 Module

Add intelligence to a simple analog signal. Flow rates are displayed in actual volume units, not merely a percent of full scale. Any linear 4-20 mA input can be characterized by using the 780. The information can be stored and retrieved for later analysis.

## Integrated Water Monitoring

Isco 6712 Samplers feature “plug and play” connection with SDI-12 compatible measuring devices - including multi-parameter sondes from leading manufacturers. Combined with the 6712's standard 512 KB of memory, enough for more than

200,000 stored readings. SDI-12 networking gives you great flexibility for logging environmental data, and for “smart sampling” event notification, triggered on any combination of up to 16 inputs.





# Isco 6712 Full-size Portable Sampler Specifications

Sampler			Controller		
Height	27.0 in.	68.6 cm	Weight	13 lbs.	5.9 kg
Diameter	20 in.	50.7 cm	Dimensions	10.3 x 12.5 x 10 in.	26 x 31.7 x 25.4 cm
Weight (Dry/Less Battery)	32 lbs.	15 kg	Operational Temperature	32° to 120°F	0° to 49°C
Material	High-strength ABS plastic outer shell Stainless steel hardware		Enclosure Rating	NEMA 4X, 6	IP67
Power Requirements	12 VDC		Program Memory	Non-volatile flash memory	
Pump			Flow Meter Signal Requirements	5 to 15 volt DC pulse or 25 millisecond isolated contact closure.	
Intake Purge	Adjustable air purge before and after each sample.		Number of Programmable Composite Samples	1 to 999 samples or continuous sampling	
Tubing Life Indicator	Provides a warning to change pump tubing.		Real Time Clock Accuracy	1 minute per month, typical	
Intake Suction Tubing			Software		
Length	3 to 99 ft.	1 to 30 m	Sample Frequency Selection	1 minute to 99 hours 59 minutes, in 1 minute increments. Non-uniform times in minutes or clock times 1 to 9,999 flow pulses	
Material	Vinyl or Teflon® lined		Sampling Modes	Uniform time, non-uniform time, flow. <i>(Flow mode is controlled by external flow meter pulses.)</i>	
Inside Dimension	¾ in.	1 cm	Programmable Sample Volumes	10 to 9,990 ml in 1 ml increments	
Pump Tubing Life	Typically 1,000,000 pump counts		Sample Retries	If no sample is detected, up to 3 attempts; user selectable	
Maximum Suction Lift	28 ft.	8.5 m	Rinse Cycles	Automatic rinsing of suction line up to 3 rinses for each sample collection	
Typical Repeatability	±5 ml or ±5% of the average volume in a set		Program Storage	5 sampling programs	
Typical Line Transport Velocity at head heights of:			Sampling Stop/Resume	Up to 24 real time/date sample stop/resume commands	
3 ft. (0.9 m)	3.0 ft./s	0.91 m/s	Controller Diagnostics	Tests for RAM, ROM, pump display, and distributor	
10 ft. (3.1 m)	2.9 ft./s	0.87 m/s			
15 ft. (4.6 m)	2.7 ft./s	0.83 m/s			
Liquid Presence Detector	Non-wetted, non-conductive sensor detects when liquid sample reaches the pump to automatically compensate for changes in head heights.				

## Ordering Information

Description	Part Number
<b>6712 Portable Sampler, Full-size</b> Includes controller with 512 KB RAM, top cover, center section, base, distributor arm, instruction manual, pocket guide.	<b>68-6710-070</b>
<b>6712 Portable Sampler with Jumbo Base</b> (as described above)	<b>68-6710-082</b>

Note: Power source, bottle configuration, suction line, and strainer must be ordered separately. Other options and accessories are also available. Contact Isco or your Isco Representative for complete information.



The 6712 Controller is an SDI-12 logger. Manual pump operations are now located on the front panel keys.



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Fax: (402) 465-3022  
E-Mail: info@isco.com

**ATTACHMENT 2**

**ISCO 720 SUBMERGED PROBE FLOW MODULE**

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# Isco 720 Submerged Probe Flow Module

The probe is mounted at the bottom of the channel, and uses a differential pressure transducer to measure the level of the flow stream. The 6700 Series or Avalanche Sampler then converts this depth measurement into flow rate. The probe's venting system automatically compensates for changes in atmospheric pressure to maintain accuracy. The 720 provides accurate measurement at sites where wind, steam, foam, turbulence, or air temperature fluctuations exist. The probe is suitable for small channels, and it accurately senses pressure even when covered with silt and sand.

Isco submerged probes are UL Classified for use in Class I, Division 1, Groups A, B, C, & D hazardous locations when installed using an Isco Intrinsically Safe Barrier and Quick-disconnect Box. This makes the submerged probe suitable in applications where flammable gases or vapors may be present.

Isco mounting rings make it easy to install the probe in round pipes, manhole inverts, and other open channels. And with the Isco Street Level Installation Tool, you can install your monitoring system from ground level, eliminating the costs and hazards of entering manholes. Most flumes are also available with an integral recess for mounting an Isco Submerged Probe.



*Simply plug in one of the environmentally-sealed modules to expand monitoring capabilities. They can easily be added or changed in the field.*



## Applications

- ◆ Flow measurement where wind, steam, foam, or turbulence exist
- ◆ Trigger sampling based on flow or level
- ◆ Flow-proportioned sample collection
- ◆ Treatment-capacity analysis
- ◆ Stormwater monitoring
- ◆ Combined sewer overflow studies
- ◆ Long-term river and stream gauging

## Standard Features

- ◆ Submerged probe accuracy unaffected by wind, steam, foam, turbulence, or air temperature change
- ◆ Built in flow conversions for most applications, including weirs and flumes, Manning formula, data points, or equation for special situations
- ◆ During program operation, current flow and level values are viewable on the sampler's LCD display
- ◆ All level data stored in the sampler is available for later retrieval, reporting, and graphing using Isco Flowlink® software

## Specifications

720 Module							
Size (H x W x D)	4.9 x 5.7 x 2.0 in	12.4 x 14.5 x 5.1 cm	Level Measurement Method	Submerged pressure transducer mounted in the flow stream			
Weight	0.9 lbs	0.4 kg					
Material	Polystyrene		Transducer Type	Differential linear integrated circuit pressure transducer			
Enclosure	NEMA 4X, 6	IP67	Level Measurement Range	Standard range probe Extended range probe	0.1 to 10 ft 0.1 to 30 ft	0.03 to 3.05 m 0.03 to 9.14 m	
Power (provided by 6700 Series Sampler)	9 to 14V DC						
Program Memory	Non-volatile, programmable flash; can be updated via interrogator port on 6700 Series Sampler using a PC		Maximum Allowable Level	Standard range probe Extended range probe	20 ft 40 ft	6.1 m 12.2 m	
Level Measurement Data Storage Interval (programmable through 6700 Series Sampler)	1, 2, 5, 10, 15, or 30 minutes		Level Measurement Accuracy				
Operating Temperature	32° to 120°F	0° to 49°C	Non-linearity, repeatability, and hysteresis at 77°F (25°C) (does not include temperature coefficient)	Level*	Error	Level*	Error
Storage Temperature	0° to 140°F	-18° to 60°C					
Submerged Probe			Extended range probe	0.033 to 5.0 ft >5.0 ft	±0.008 ft/ft ±0.012 ft/ft	0.01 to 1.52 m >1.52	±0.008 m ±0.012 m
Hazardous Location Rating	UL Classified for use in Class I, Division 1, Groups A, B, C, & D hazardous locations as defined by Article 500 of the National Electrical Code when installed with Isco Intrinsically Safe Barrier and Quick-disconnect Box per control drawing 60-3403-131.			0.1 to 15 ft 0.1 to 21 ft 0.1 to 30 ft	±0.01 ft ±0.03 ft ±0.3 ft	0.03 to 4.6 m 0.03 to 6.4 m 0.03 to 9.1 m	±0.006 m ±0.009 m ±0.091 m
Length (with standard tip)	9.5 in	24.1 cm	Temperature Coefficient	Level*	Error	Level*	Error
Diameter	0.875 in	2.2 cm					
Frontal Area	0.601 in <sup>2</sup>	3.88 cm <sup>2</sup>	Standard range probe	0.1 to 4.0 ft 4.0 to 10 ft	±0.005 ft/°F ±0.007 ft/°F	0.03 to 1.22 m 1.22 to 3.05 m	±0.0027 m/°C ±0.0038 m/°C
Cable Length			Extended range probe	0.1 to 30 ft	±0.008 ft/°F	0.03 to 9.14 m	±0.0044 m/°C
Standard range probe	25 ft	7.6 m					
Extended range probe	50 ft	15.2 m	Operating Temperature	32° to 120°F		0° to 49°C	
Cable Diameter	0.3 in	0.8 cm	Compensated Temperature	32° to 100°F		0° to 38°C	
Weight (including cable)			Materials	Type 316 stainless steel, chlorinated polyvinyl chloride (CPVC) Polyvinyl chloride (PVC)			
Standard range probe	3 lbs	1.4 kg	Submerged probe				
Extended range probe	7 lbs	3.2 kg	Cable				
			*Actual vertical distance between the submerged probe and the liquid surface.				

## Ordering Information

Description	Part Number
<b>720 Submerged Probe Flow Module</b>	
with 10 ft (3.05m) level measurement range	68-6700-068
with 30 ft (9.14 m) level measurement range	68-6700-069
<b>720 Accessories</b>	
Quick-disconnect Box	60-3224-003
Intrinsically Safe Barrier	60-3404-060



### Teledyne Isco, Inc.

4700 Superior Street  
Lincoln NE 68504 USA  
Phone: (402) 464-0231  
USA and Canada: (800) 228-4373  
Fax: (402) 465-3022  
E-Mail: [info@isco.com](mailto:info@isco.com)  
Internet: [www.isco.com](http://www.isco.com)

Libby Superfund Site Operable Unit 3 Standard Operating Procedure
---

Date: 03/02/2011

OU3 SOP 19 (Rev. 0)

Title: STREAM POOL CLASSIFICATION AND CHARACTERIZATION

**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager

\_\_\_\_\_

\_\_\_\_\_

SOP Author

\_\_\_\_\_

\_\_\_\_\_

Revision Number	Date	Reason for Revision
0	03/02/2011	--

## 1.0 INTRODUCTION

The nature and availability of pools are key habitat metrics for Cutthroat and Rainbow trout, especially in small streams and especially during the summer months. This standard operating procedure (SOP) is for use at the Libby Asbestos Superfund Site OU3. This SOP describes methods used for classifying and characterizing pools in streams in OU3. This protocol does not cover lakes, reservoirs or large, non-wadeable rivers. This protocol is based on Hickman (1982), Raleigh (1984), and Lewis (1969), and provides the information needed to rate the pool class parameter ( $V_{15}$ ) for the Habitat Suitability Index (HSI) models for both Cutthroat and rainbow trout (Hickman 1982, Raleigh 1984).

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in field work in OU3 must follow health and safety protocols described in the appropriate health and safety plan. Inhalation exposure to asbestos during sampling may increase the risk of lung cancer, mesothelioma, asbestosis, and other respiratory diseases. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan.

## 3.0 DEFINITIONS

**Pool Class.** Table 1 provides pool class definitions are based on the USFWS HSI models for rainbow trout and Cutthroat trout (Hickman and Raleigh 1982, Raleigh et al 1984, Lewis 1969). A slight adjustment to optimal pool depth for Class 1 (lowering from  $>1.5\text{m}$  to  $\geq 1.0\text{m}$ ) is based on Adams et. al. (2008). The addition of specific pool depths to Class 2 and 3 is based on Harig and Fausch (2002).

**Pool Surface Area Fraction:** The surface area fraction of different pool classes within a stream reach determines the Pool Class Rating for that stream reach, i.e., variable  $V_{15}$  for the salmonid HSI models.

Stream reaches will be rated as A, B or C as follows:

- A:  $\geq 30\%$  of the reach is comprised of 1st class pools
- B:  $\geq 10\%$  to  $< 30\%$  1st class pools, or  $\geq 50\%$  2nd class pools
- C:  $< 10\%$  1st class pools and  $< 50\%$  2nd class pools

#### **4.0 RESPONSIBILITIES**

This section presents a brief definition of field roles, and the responsibilities generally associated with them.

**Field Supervisor:** Responsible for planning and overseeing the implementation of field survey activities. Ensures that all work is performed in accord with applicable SOPs, and for notifying EPA of any deviations.

**Survey Technician:** Assists the Field Supervisor perform the survey by collecting and recording pool habitat data in accord with this SOP.

#### **5.0 EQUIPMENT**

- Field notebook - a bound book used to record progress of sampling effort and record any problems and field observations during sampling.
- Three-ring binder book- to store necessary forms used to record and track samples collected at the site. Binders will contain the Pool Classification and Characterization Field Sampling Data Sheet (FSDS)
- Permanent marking pen - used to record information in field logbooks and data sheets.
- Global Positioning System (GPS) unit
- Marking stakes
- Digital Camera
- 100 m measuring tapes
- Rigid meter sticks for measuring pool dimensions in meters

#### **6.0 STEAM POOL CLASSIFICATION AND AREA FRACTION PROCEDURES**

##### **6.1 Sampling Reach**

Locate the stream reach of interest per directions provided in the sampling and analysis plan (SAP). On the Pool Classification and Characterization FSDS, record the length and average width of the stream reach in meters. On the back of the sheet, provide a sketch of the reach that illustrates stream curves and any major vegetation features, snags or boulders that create cover.

##### **6.2 Pool Classification**

The overall objective of this effort is to identify the nature and availability of pools within the stream reach. Table 1 provides a method for characterizing the pools so that the data can be used to rate the reach and use this parameter in an HSI model. Start at the bottom of the reach



and record the information indicated on the Pool Classification and Characterization FSDS for each pool observed. Marking stakes may be useful to keep track of progress along the reach. Note that in some cases it may be necessary to probe the bottom with the yardstick to identify pool locations.

Since pools will have irregular shapes, for the length and width measurements, measure the longest and widest part of the pool in meters. Do not count pools that have a surface area  $<0.15 \text{ m}^2$ . For depth, measure the deepest part of the pool in meters. Do not count pools  $<0.2 \text{ m}$  in depth.

The percent cover is evaluated based on the ability to view the bottom of the pool. The value recorded on the FSDS is the percent of the pool bottom that is obscured. There are many factors that could obscure the bottom of a pool, including depth, surface turbulence, or the presence of structures such as logs, debris, snags, boulders, or overhanging banks and vegetation.

**Note that the shaded areas on the Pool Classification and Frequency FSDS can be completed later.**

## **7.0 REFERENCES**

Adams, P., C. James and C. Speas. 2008. Rainbow trout (*Oncorhynchus mykiss*) Species and Conservation Assessment. Prepared for the Grand Mesa, Uncompahgre and Gunnison National Forests.

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Harig, A. and K. Fausch. 2002. Minimum habitat requirements for established translocated Cutthroat trout populations. *Ecological Applications* 12(2) 535-551.

Hickman, T. and R. F. Raleigh. 1982. Habitat suitability index models: Cutthroat trout. United States Fish and Wildlife Service Report FWS/OBS-82/10.5, Fort Collins, CO.

Lewis, S.L. 1969. Physical factors influencing fish populations in pools of a trout stream. *Trans. Am. Fish. Soc.* 92(2):140-145.

Raleigh, R.F. et al. 1984. Habitat suitability information: Rainbow trout. United States Fish and Wildlife Service. FWS/OBS-82/10.60, Fort Collins, CO.

**TABLE 1**  
**POOL CLASS DEFINITIONS**

<b>Pool Class</b>	<b>Description</b>
<b>1st</b>	Large and deep. Pool depth and size are sufficient to provide a low velocity resting area for several adult fish. More than 30 percent of the pool bottom is obscured due to depth, surface turbulence, or the presence of structures, for example, logs, debris, boulders, or overhanging banks and vegetation. The pool depth is $\geq 1.0\text{m}$ deep (in streams $< 5\text{m}$ wide). Rainy Creek averages $< 2\text{m}$ in width.
<b>2nd</b>	Moderate size and depth. Pool depth and size are sufficient to provide a low velocity resting area for a few adult fish. From 5 to 30 percent of the pool bottom is obscured due to depth, surface turbulence, or structures. Typical second class pools are large eddies behind boulders and low velocity moderately deep areas beneath overhanging banks and vegetation. Pool depth may range from 0.3 to $< 1.0\text{m}$ .
<b>3rd</b>	Small or shallow or both. Pool depth and size are sufficient to provide a low velocity resting area for one or two adult fish. Cover, if present, is in the form of shade, surface turbulence, or very limited structure. Typical third class pools are wide, shallow pool areas of streams or small eddies behind boulders. Virtually the entire bottom area is discernable. Pool depth is $< 0.3\text{m}$ to $0.2\text{m}$ .

Sources:

Adams et. al. (2008)  
 Harig and Fausch (2002)  
 Hickman and Raleigh 1982  
 Lewis (1969)  
 Raleigh et al. (1984)

**ATTACHMENT A**

**POOL CLASSIFICATION AND CHARACTERIZATION  
FIELD SAMPLING DATA SHEET**

## LIBBY OU3 FIELD SAMPLE DATA SHEET

### POOL CLASSIFICATION AND FREQUENCY

Form Completed By: \_\_\_\_\_

Logbook Page No: \_\_\_\_\_

Date: \_\_\_\_\_

Time: \_\_\_\_\_

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

Top of reach:      X coord: \_\_\_\_\_      Y coord: \_\_\_\_\_

Bottom of reach: X coord: \_\_\_\_\_ Y coord: \_\_\_\_\_

[illegible]

*Shaded areas do not have to be calculated in the field.*

**LIBBY OU3 FIELD SAMPLE DATA SHEET  
POOL CLASSIFICATION AND FREQUENCY**

**Sketch**

<b>Pool Class</b>	<b>N Total</b>	<b>% Area</b>	<b>% Reach</b>
1 <sup>st</sup>			
2 <sup>nd</sup>			
3 <sup>rd</sup>			

<i>Select one of the following:</i>		
<b>Rating</b>		<b>Description</b>
	A	≥ 30% of the reach is comprised of 1 <sup>st</sup> class pools
	B	≥ 10% to < 30% 1 <sup>st</sup> class pools, or ≥ 50% 2 <sup>nd</sup> class pools
	C	< 10% 1 <sup>st</sup> class pools and < 50% 2 <sup>nd</sup> class pools

Libby Superfund Site Operable Unit 3 Standard Operating Procedure
---

Date: 03/02/2011

OU3 SOP 20 (Rev. 0)

Title: SURFACE WATER TEMPERATURE DATA LOGGER

**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager

\_\_\_\_\_

\_\_\_\_\_

SOP Author

\_\_\_\_\_

\_\_\_\_\_

Revision Number	Date	Reason for Revision
0	03/02/2011	--

## **1.0 INTRODUCTION**

Water temperature is a key habitat metric for Cutthroat and Rainbow trout, especially in small streams and especially during the summer months. This standard operating procedure (SOP) is for use at the Libby Asbestos Superfund Site OU3. This SOP describes methods and equipment commonly used for collecting continuous surface water temperature. The purpose of this protocol is to describe guidelines for the placement, retrieval and documentation of temperature data loggers at individual stream sites in OU3. This protocol does not cover lakes, reservoirs or large, non-wadeable rivers. This protocol is based on Zaroban (2000).

## **2.0 HEALTH AND SAFETY WARNING**

All personnel engaged in surface water temperature sampling must follow health and safety protocols described in the OU3 health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## **3.0 DEFINITIONS**

**Temperature Data Logger:** Temperature data loggers are the preferred method for collecting continuous temperature data records. There are many manufacturers and models of data loggers from which to choose. At present, StowAway, TidbiT and HOBO Water Temp Pro by Onset Computer Corporation are more commonly used models. The following characteristics are recommended:

1. Submersible, waterproof logger
2. Accuracy  $\pm 0.2^{\circ}\text{C}$
3. Measurement range  $-4^{\circ}$  to  $37^{\circ}\text{C}$  ( $24^{\circ}$  to  $99^{\circ}\text{F}$ )
4. Resolution  $< 0.2^{\circ}\text{C}$  (needs to be less than accuracy)
5. Programmable start time/date
6. User-selectable sampling interval

Other issues to consider when selecting a data logger are memory capacity and battery life. The storage capacity needed will depend on the sampling interval (i.e. 30 seconds, 15 minutes, 2 hours) and how long the logger will be deployed (i.e. 7 days, 6 months, 1 year). See sampling methods in Section 5.0. For battery life, some loggers have factory replaceable batteries and others have non-replaceable batteries which should last 5 years with typical use.

**Software, Connector Cable and Shuttle:** The appropriate data logger software and a connector cable from a computer to the data logger is also needed, along with a shuttle which allows downloading the data in the field. Periodically downloading the data in the field reduces the risk of losing significant amounts of data.

#### **4.0 RESPONSIBILITIES**

This section presents a brief definition of field roles, and the responsibilities generally associated with them.

**Project Manager:** Selects site-specific field sampling program with input from other key project staff, and applicable oversight agencies.

**Sampling Technician (or other designated personnel):** Assists the PM in the implementation of tasks.

### **5.0 SURFACE WATER TEMPERATURE DATA LOGGER PROCEDURES**

#### **5.1 Calibration**

It is important to check the accuracy of the data logger(s) before and after field deployment. The manufacturer's instructions should be followed during calibration. This section contains an overview of a typical calibration.

The accuracy of the temperature data logger should be tested in a water bath at two temperatures: 0°C and 20°C. A NIST (National Institute of Standards and Technology) traceable thermometer accurate to 0.2°C is preferred to determine accuracy.



The first step is programming the logger. Connect the logger to a computer and set the measurement interval and duration, measurement units, start date and time. Program the logger to record data at a short recording interval (five minutes) to reduce calibration time. Be sure the clocks in the computer, logger and the clock used during the calibration procedure are consistent.

The next step is preparing two water baths. One bath should be an ice water bath in a large cooler or other covered and insulated container. The second bath should be held at room temperature. Verify that each bath is uniform temperature (mixing may be required). Place the loggers in one of the baths long enough to equilibrate to the temperature of the bath. After the equilibration period, measure and record instantaneous water bath temperatures with the NIST thermometer as close to the time the logger is recording a measurement value as possible. Once the water bath temperature measurements have been recorded, place the loggers in the second bath and repeat the process. A calibration form is included in Attachment A.

The last step is comparing the logger data to the instantaneous thermometer data. Once the measurements have been recorded for both temperatures, remove the loggers from the water bath, connect to a computer then download and display the data. Compare the logger data to the NIST thermometer data recorded on the calibration form and document each pair of readings and the magnitude of any discrepancy. Loggers are acceptable for placement only if their accuracy is within  $\pm 0.3^{\circ}\text{C}$ . A logger that exceeds the  $\pm 0.3^{\circ}\text{C}$  tolerance limit should be returned to the manufacturer for adjustment or replacement.

## **5.2 Surface Water Temperature Data Logger Placement Procedures**

### **Launch Logger**

Launch the logger according the manufacturer's recommendations. Program the logger to collect data at intervals appropriate for program goals and to record temperature measurements in degrees Celsius.

### **Site Selection**

The stream and reach selected for logger placement is determined by the goals and objectives of the monitoring activity. In addition, the specific location within the reach, e.g., the deepest pool, is determined by the goals and objectives.

### **Logger Placement**

Once at the site and in the stream channel, find a shaded spot where the water is adequately mixed and not influenced by localized warm or cool water sources such as ground water, point sources, or direct sunlight. Verify that the site is well-mixed horizontally and vertically in the stream or pool with instantaneous temperature measurements using a calibrated handheld thermometer. Record these findings for each temperature logger site in a field log book. If the logger does not come with a protective case, it should be placed in a housing to protect the equipment from natural, wildlife or human disturbance.

### **Documentation**

A description of the site should be recorded in the field log book. The description should, at a minimum, include location identification (sampling location), latitude and longitude, instantaneous water temperature, pool width and depth, date and time of the actual placement, and logger serial number.

### **Maintenance**

Whenever feasible, it is recommended to visit the site monthly to make a calibration check and any needed adjustment to the logger. When a visit is made, record the date, time and instantaneous water temperature. Check the security of the housing and adjust if necessary. Remove debris or sediment buildup.

### **5.3 Surface Water Temperature Data Logger Retrieval Procedures**

#### **Remove Logger**

At the completion of the monitoring objective, remove the logger from the site. In the field log book, document the condition of the site and the logger. At a minimum, record such things as whether the logger is still in the water and any signs of vandalism or disturbance. Record the date, time and instantaneous water temperature at the time of retrieval. Remove any other equipment from the site, if applicable.

#### **Download Data**

Depending on the type of logger used, data may be downloaded periodically in the field with a shuttle. If not, the logger should be connected to a computer and downloaded using the manufacturer's procedures for the data logger type. Once the data have been downloaded by the data logger software package the data can be exported into Microsoft Excel and/or txt files.

### **6.0 DECONTAMINATION**

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in Libby OU3 SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan.

### **7.0 REFERENCES**

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Zaroban, D.W. 2000. Protocol for Placement and Retrieval of Temperature Data Loggers in Idaho Streams. Idaho Division of Environmental Quality, Boise, Idaho.

**Attachment A**

**Temperature Logger Calibration Form**

# Temperature Logger Calibration Form

# Logger

Manufacturer \_\_\_\_\_ Model \_\_\_\_\_ Serial Number \_\_\_\_\_

Calibration Date \_\_\_\_\_ Conducted by \_\_\_\_\_

# NIST Certified Thermometer

Manufacturer \_\_\_\_\_ Model \_\_\_\_\_ Serial Number \_\_\_\_\_

[illegible]

**ATTACHMENT B**

**LIBBY-SPECIFIC LABORATORY MODIFICATIONS**

LB-000016  
LB-000019  
LB-000028  
LB-000029B  
LB-000030  
LB-000066C

*This page intentionally left blank to facilitate double-sided printing.*



**Request for Modification**  
To  
**Laboratory Activities**  
LB-000016

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**  
**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: \_\_\_\_\_

Requester: Jeanne Orr Title: President  
Company: Reservoirs Environmental, Inc. Date: December 2, 2002

**Description of Modification:**

Permanent modifications and clarifications to the Transmission Electron Microscopy analysis of air samples using ISO 10312. The purpose of the attached is to document permanent historic modifications & clarifications.

**Reason for Modification:**

To optimize the efficiency of air sample analysis and to provide consistency in analytical procedures and data recording in the project laboratories.

**Potential Implications of this Modification:**

Modifications reflect changes necessary to clarify ISO requirements in relation to project-specific issues. No negative implications to these modifications are anticipated. Positive implications are consistency in procedures between and within project laboratories and documentation of those procedures.

Laboratory Applicability (circle one): ☒ All Individual(s) \_\_\_\_\_

**Duration of Modification (circle one):**

Temporary Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

☒ Permanent (complete Proposed Modification Section) Effective Date: HISTORIC

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by TEM analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Please see the attached for the description of the TEM-ISO clarifications/modifications

Technical Review: R.K. Mahoney, ENEC Date: 23 April 2003  
(Laboratory Manager or designate)

Project Review and Approval: [Signature] Date: 4 April 2003  
(Volpe: Mark Raney)

Approved By: Jane Goldade Title: Project Checklist Date: 3 April 2003  
(USEPA: Mary Goldade)



**1. Modification:**

The ISO method requirement is if the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. A rejection criteria of >25% obscuration and <50% intact grid openings will be used for this project. The 25 % overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, Camp Dresser McKee, Volpe Center, and Reservoirs.

**2. Modification:**

ISO 10312 is a direct preparation method. If samples are visibly overloaded or contain loose debris and they have not been previously analyzed (the filter is whole) they will be prepared indirectly according to procedures described in ASTM D5755-95. If the sample has been previously analyzed or rejected in the microscope (section removed from the filter), prepare the sample indirectly according to EPA/540/2-90/005a by plasma ashing a portion of the original filter and depositing an aliquot on a secondary filter. Secondary filters will be analyzed according to the ISO counting rules for this project. Calculations are modified to contain a dilution factor. This indirect preparation procedure is embraced to enable the capture of data from samples that otherwise would be rejected.

**3. Clarification:**

Stopping rules for ISO analyses are completion of the grid opening on which the 100<sup>th</sup> asbestos structure has been recorded, or a minimum of four grid openings. For this project, a maximum of ten grid openings will be read unless specifically instructed otherwise.

If abundant chrysotile is present, the chrysotile count may be terminated at the end of the grid opening where the 100<sup>th</sup> chrysotile structure is counted. The analysis will continue recording amphibole fibers only until the remaining grid openings to be analyzed are completed. The grid opening location designation will be followed by a "\*" to indicate the grid openings where only amphibole asbestos was recorded, i.e. K6\*.

This clarification in structure counting and recording is to provide consistency in analytical procedures and data recording in the project laboratories.

**4. Modifications and clarifications: Structure counting and recording**

- a. **Modification:** Non-asbestos structures are not being recorded. This project-specific modification stems from our need only to quantify contaminants of concern: the asbestos levels at a given sample location.
- b. **Modification:** The overall dimensions of disperse clusters (CD) and disperse matrices (MD) will not be recorded in two perpendicular directions. The matrix type and individual structures associated with the matrix or cluster will be recorded as described in the ISO method.
- c. **Modification:** Structures that intersect a non-countable grid bar will be recorded on the count sheet but excluded from the structure density and concentration calculations.
- d. **Modification:** If a structure originates in one grid opening and extends into an adjacent grid opening, providing that it does not intersect a non-counting grid bar, the entire length of the fiber is recorded.
- e. **Clarification:** If a structure intersects both a countable and a non-countable grid bar, the observed length of the structure will be recorded.

These modifications and clarifications in structure counting and recording are to provide consistency in analytical procedures and data recording in the project laboratories.

**Mahoney, Ron**

---

**From:** Raney, Mark [RANEY@VOLPE.DOT.GOV]  
**Sent:** Tuesday, April 22, 2003 11:09 AM  
**To:** 'Mahoney, Ron'  
**Subject:** FW: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017



LB-000015\_rev (MR)  
4-4-03 email...



LB-000016\_rev (MR)  
4-4-03 email...



LB-000017\_rev (MR)  
4-4-03 email...

FYI

> -----Original Message-----

> From: Raney, Mark  
> Sent: Friday, April 04, 2003 9:31 AM  
> To: 'Beckham, Richard'; 'Goldade.mary@EPAMail.epa.gov'; 'mgoldade@peakpeak.com'  
> Cc: Autio, Anni  
> Subject: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017

>  
> Volpe provides approval to revised MODs LB-000015, LB-000016, & LB-000017 as attached. The attached MODs include the following changes to the previous versions (received 4/1/03).

>  
> \* The date indicated in the "Effective Date" field was removed and replaced with "HISTORIC"  
> \* Under the "Description of Modification" section the following sentence was added "The purpose of the attached is to document permanent historic modifications & clarifications."

>  
> If you have any questions as to these changes or the reason behind them let me know. Please proceed with distribution of the accepted versions of the attached for final hardcopy signature.

>  
> Mark.

>  
> <<LB-000015\_rev (MR 4-4-03 email).doc>> > <<LB-000016\_rev (MR 4-4-03 email).doc>> > <<LB-000017\_rev (MR 4-4-03 email).doc>>

> -----Original Message-----

> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]  
> Sent: Tuesday, April 01, 2003 10:47 AM  
> To: 'Goldade.mary@EPAMail.epa.gov'; 'RANEY@VOLPE.DOT.GOV';  
> 'mgoldade@peakpeak.com'  
> Cc: Autio, Anni  
> Subject: FW: LB-000015, LB-000016, and LB-000017

>  
> For your review and approval.

>  
> - Richard Beckham

> -----Original Message-----

> From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]  
> Sent: Monday, March 31, 2003 6:11 PM  
> To: Beckham, Richard  
> Subject: LB-000015, LB-000016, and LB-000017

>  
> Richard,

>  
> These should be final. The only recent revision is the addition of the  
> Effective Date. These need to go to Mark and Mary for their final blessing.

> <<LB-000015(rev 3\_31\_03).doc>> <<LB-000016 rev. (3\_31\_03).doc>>  
> <<LB-000017 rev(3\_31\_03).doc>>  
>  
> R.K. Mahoney  
> Senior Analyst  
> Special Projects Coordinator  
> EMSL Analytical, Inc.  
> Westmont, NJ  
> 800.220.3675, x1218  
> rmahoney@emsl.com  
>  
> << File: LB-000015(rev 3\_31\_03).doc >> << File: LB-000016 rev. (3\_31\_03).doc >> << File: LB-000017 rev(3\_31\_03).doc >>

**Mahoney, Ron**

---

**From:** Raney, Mark [RANEY@VOLPE.DOT.GOV]  
**Sent:** Wednesday, April 23, 2003 9:02 AM  
**To:** 'Mahoney, Ron'  
**Subject:** FW: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017



LB-000015(rev  
3\_31\_03).doc



LB-000016 rev.  
(3\_31\_03).doc



LB-000017  
rev(3\_31\_03).doc

Ron,

I almost forgot to forward you this....

See Mary's earlier email below, regarding EPA's approval for MODs LB-15, 16, & 17.

Let me know if you have any questions.

Mark.

-----Original Message-----

**From:** Goldade, Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]  
**Sent:** Thursday, April 03, 2003 5:49 PM  
**To:** Beckham, Richard  
**Cc:** Autio, Anni; 'mgoldade@peakpeak.com'; 'RANEY@VOLPE.DOT.GOV'  
**Subject:** EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017

Richard,  
Mark will modify LB-000015, 16 & 17 to indicate that the Effective Date  
is: Historical.  
EPA approves these mods with this changed completed.

"Beckham,  
Richard" To: Mary Goldade/EPR/R8/USEPA/US@EPA, "RANEY@VOLPE.DOT.GOV"  
<BeckhamRE@cdm.co <RANEY@VOLPE.DOT.GOV>, "mgoldade@peakpeak.com"  
<mgoldade@peakpeak.com>  
m> cc: "Autio, Anni" <AutioAH@cdm.com>  
Subject: FW: LB-000015, LB-000016, and LB-000017  
04/01/03 08:47 AM

For your review and approval.

- Richard Beckham

-----Original Message-----

**From:** Mahoney, Ron [mailto:Rmahoney@EMSL.com]  
**Sent:** Monday, March 31, 2003 6:11 PM  
**To:** Beckham, Richard

Subject: LB-000015, LB-000016, and LB-000017

Richard,

These should be final. The only recent revision is the addition of the Effective Date. These need to go to Mark and Mary for their final blessing.

<<LB-000015(rev 3\_31\_03).doc>> <<LB-000016 rev. (3\_31\_03).doc>>  
<<LB-000017 rev(3\_31\_03).doc>>

R.K. Mahoney  
Senior Analyst  
Special Projects Coordinator  
EMSL Analytical, Inc.  
Westmont, NJ  
800.220.3675, x1218  
rmahoney@emsl.com

(See attached file: LB-000015(rev 3\_31\_03).doc)(See attached file:  
LB-000016 rev. (3\_31\_03).doc)(See attached file: LB-000017  
rev(3\_31\_03).doc)



## Request for Modification

To  
Laboratory Activities

LB-000019

**Instructions to Requester:** E-mail form to contacts at bottom of form for review and approval.

**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney

Title: Senior Analyst/Special Projects Coordinator

Company: EMSL Analytical, Inc.

Date: 21 January 2003

Description of Modification:

Clarification of bench sheet recording format for grid openings in which no countable structures are recorded.

Reason for Modification:

The electronically deliverable spread sheet for TEM analysis developed for the Libby project requires "ND" (None Detected) to be entered for grid openings in which no countable structures are recorded. The ND code has been used on all electronic deliverables for the Libby project. The code "NSD" (No Structure Detected) has been used on hand written bench sheets up until this date. As of 21 January 2003, "ND" will be used on the bench sheets as well as the electronically deliverables.

Potential Implications of this Modification:

There are no potential negative implications resulting from this clarification of terms.

Laboratory Applicability (circle one): All Individual(s) EMSL Analytical, Inc.

Duration of Modification (circle one):

Temporary Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent

(Complete Proposed Modification Section)

Effective Date: 21 January 2003

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney  
(Laboratory Manager or designate)

Date: 27 March 2003

Project Review and Approval: Mark Raney

(Volpe: Mark Raney)

Date: 7 March 2003

Approved By: Mary Goldade

Date: 7 March 2003

Title: EPA Regional Chemist

(USEPS: Mary Goldade)

**Mahoney, Ron**

---

**From:** Raney, Mark [RANEY@VOLPE.DOT.GOV]  
**Sent:** Friday, March 07, 2003 2:50 PM  
**To:** 'Beckham, Richard'; 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; Raney, Mark; 'brattin@syrres.com'; 'Goldade.mary@EPAMail.epa.gov'; Montera, Jeff  
**Subject:** RE: MOD LB-000019

I find Laboratory Request for Modification # LB-000019 acceptable as written and here by provide Volpe approval to this MOD.

Richard, Please make sure MOD ID#s get inserted onto the mod forms themselves (not just the file ID), so you will be able to identify the IDs based upon hardcopy alone. Also, even though this MOD is applicable to an individual lab, all MODs are to be forwarded to all labs for informational purposes and to give them an opportunity to provide comments. All labs however are REQUIRED to provide comments to only MODs that are applicable to all labs.

Mark Raney  
Environmental Engineer

US DOT / Volpe Center  
Environmental Engineering Division, DTS-33  
phone: 617-494-2377  
cell: 617-694-8223  
fax: 617-494-2789  
raney@volpe.dot.gov

-----Original Message-----

**From:** Beckham, Richard [mailto:BeckhamRE@cdm.com]  
**Sent:** Thursday, March 06, 2003 9:54 AM  
**To:** 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; 'Raney@volpe.dot.gov'; 'brattin@syrres.com'; 'Goldade.mary@EPAMail.epa.gov'; Montera, Jeff  
**Subject:** MOD LB-000019

This MOD impacts only EMSL. For your review and comment:

<<LB-000019.doc>>  
- Richard Beckham

---

**Mahoney, Ron**

---

**From:** Mary Goldade [mgoldade@peakpeak.com]  
**Sent:** Friday, March 07, 2003 12:29 PM  
**To:** Raney, Mark  
**Cc:** Jeff G. Montera; rmahoney@emsl.com; Autio, Anni; William Brattin; Goldade.Mary@epamail.epa.gov  
**Subject:** Re: MOD LB-000019

I agree that this mod form is acceptable, and should be discussed on the next lab call to be certain similar issues are not encountered at other labs.

Mary

----- Original Message -----

**From:** "Raney, Mark" <RANEY@VOLPE.DOT.GOV>  
**To:** "'Goldade, Mary (HOME)'" <mgoldade@peakpeak.com>  
**Sent:** Friday, March 07, 2003 10:18 AM  
**Subject:** FW: MOD LB-000019

>  
> FYI  
>  
>  
> -----Original Message-----  
> **From:** Beckham, Richard [mailto:BeckhamRE@cdm.com]  
> **Sent:** Thursday, March 06, 2003 9:54 AM  
> **To:** 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio,  
> Anni; 'Raney@volpe.dot.gov'; 'brattin@syrres.com';  
> 'Goldade.mary@EPAMail.epa.gov'; Montera, Jeff  
> **Subject:** MOD LB-000019  
>  
>  
> This MOD impacts only EMSL. For your review and comment:  
>  
> <<LB-000019.doc>>  
> - Richard Beckham  
>  
>  
>



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## Request for Modification

To

Laboratory Activities

LB-000028

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**

**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney Title: Senior Analyst / Special Projects Coordinator  
Company: EMSL Analytical, Inc. Date: 17 June 2003

### Description of Modification:

This is a clarification pertaining to the re-analysis of TEM samples when some of the originally read grid openings in a sample selected for re-analysis have become unreadable. In the event that more than half of the originally read grid openings have become unreadable, select the closest adjacent sample from the same sample delivery group with adequate intact grid openings for re-analysis. If half or less of the original openings on the sample selected are unreadable, make note in the Comments box in Data Entry 1 of the TEM EDD as to which grid openings are unreadable, and proceed with analysis of the original sample.

### Reason for Modification:

This clarification is intended to provide more complete TEM re-analysis data.

### Potential Implications of this Modification:

There are no negative implications to this clarification.

Laboratory Applicability (circle one): ☒ All Individual(s) \_\_\_\_\_

### Duration of Modification (circle one):

Temporary Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

☒ Permanent (Complete Proposed Modification Section) Effective Date: 17 June 2003

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney EMSL Date: 18 July 2003  
(Laboratory Manager or designate)

Project Review and Approval: Mark Roney Date: 7/18/03  
(Volpe: Project Technical Lead or designate)

Approved By: Walter Goldade Date: 6/24/03

Title: Project Chemist  
(USEPA: Project Chemist or designate)

Mary Goldade

06/24/03 01:20 PM

To: "Beckham, Richard" <BeckhamRE@cdm.com>

cc: "Autio, Anni" <AutioAH@cdm.com>, "Bill Egeland" <begeland@mastest.com>, "Bob.Shumate@battaenv.com" <Bob.Shumate@battaenv.com>, "brattin@syrres.com" <brattin@syrres.com>, "Charlie LaCerra" <clacerra@emsl.com>, "corbin77@atc-enviro.com" <corbin77@atc-enviro.com>, "dmazzaferro@mastest.com" <dmazzaferro@mastest.com>, "Gustavo Delgado" <gdelgado77@atc-enviro.com>, "Garth B. Freeman" <gfreeman@mastest.com>, "jeanneorr@resienv.com" <jeanneorr@resienv.com>, "mgoldade@peakpeak.com" <mgoldade@peakpeak.com>, "m\_szynskie@resienv.com" <m\_szynskie@resienv.com>, "Naresh C. Batta" <ncbatta@battaenv.com>, "Raney@volpe.dot.gov" <Raney@volpe.dot.gov>, "rdemalo@emsl.com" <rdemalo@emsl.com>, "rhatfield@mastest.com" <rhatfield@mastest.com>, "rmahoney@emsl.com" <rmahoney@emsl.com>, "Shu-Chun Su" <scsu@delanet.com>, "William Longo" <wlongo@mastest.com>

Subject: Re: EPA Approved w/ revisions MOD LB-000028

EPA approves Mod LB-000028 with revisions as attached.



LB-000028 (MG 6-24-03).

**Mary Goldade**

**Regional Superfund Chemist**



U.S. Environmental Protection Agency, Region 8

999 19<sup>th</sup> Street, Suite 300

Mail Code: BEPR-P5

Denver, CO 80202

Phone: (303) 312-7024

Fax: (303) 312-6065

email: goldade.mary@epa.gov

"Beckham, Richard" <BeckhamRE@cdm.com>



"Beckham, Richard"  
<BeckhamRE@cdm.com>

06/23/03 08:42 AM

To: "Charlie LaCerra" <clacerra@emsl.com>, "Charlie LaCerra" <clacerra@emsl.com>, "jeanneorr@resienv.com" <jeanneorr@resienv.com>, "rdemalo@emsl.com" <rdemalo@emsl.com>, "rmahoney@emsl.com" <rmahoney@emsl.com>, "William Longo" <wlongo@mastest.com>, "rhatfield@mastest.com" <rhatfield@mastest.com>, "Bill Egeland" <begeland@mastest.com>, "Bob.Shumate@battaenv.com" <Bob.Shumate@battaenv.com>, "Naresh C. Batta" <ncbatta@battaenv.com>, "Shu-Chun Su" <scsu@delanet.com>, "corbin77@atc-enviro.com" <corbin77@atc-enviro.com>, "Gustavo Delgado" <gdelgado77@atc-enviro.com>, "Garth B. Freeman" <gfreeman@mastest.com>, "Autio, Anni" <AutioAH@cdm.com>, "Raney@volpe.dot.gov" <Raney@volpe.dot.gov>, "brattin@syrres.com" <brattin@syrres.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, "dmazzaferro@mastest.com" <dmazzaferro@mastest.com>, "mgoldade@peakpeak.com" <mgoldade@peakpeak.com>, "m\_szynskie@resienv.com" <m\_szynskie@resienv.com>

cc:

Subject: MOD LB-000028

This MOD impacts all labs. For your review and comment.

- Richard Beckham

<<LB-000028.doc>>

---

**From:** "LaCerra, Charles" <CLaCerra@EMSL.com>  
**To:** "Carr, Kim" <KCarr@EMSL.com>; "EMSL Mobile Lab - Asbestos" <mobileasbestoslab@EMSL.com>  
**Sent:** Friday, July 18, 2003 5:57 AM  
**Attach:** LB-000025\_rev (MG 6-04-03 email).doc; LB-000027 (MG 6-24-03).doc; LB-000028 (MG 6-24-03).doc  
**Subject:** FW: MODs: LB-000025, 26, 27 & 28

-----Original Message-----

From: Raney, Mark [mailto:RANEY@VOLPE.DOT.GOV]  
Sent: Friday, July 18, 2003 7:53 AM  
To: 'Beckham, Richard'; Autio, Anni  
Cc: 'Goldade, Mary'; 'Goldade, Mary (HOME)'; 'Orr, Jeaane at Reservoir  
Env'; 'Mahoney, Ron'; 'Demalo, Rob (EMSL)'; 'LaCerra, Charles'  
Subject: MODs: LB-000025, 26, 27 & 28

Richard,

LB-000025 (EMSL): Volpe provided approval (with revisions) on 6/18/03 & EPA approved on 5/14/03 (see emails and attachment below). I have yet to see a final version for signature. EMSL should finalize, sign and distribute for signature.

LB-000026 (EMSL): Approved and signed by both Volpe and EPA.

LB-000027 (RESI): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). RESI should finalize, sign and distribute for signature.

LB-000028 (EMSL): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). EMSL should finalize, sign and distribute for signature.

Please let me know if anyone has any questions.

Mark.

7/18/2003

-----Original Message-----

From: Beckham, Richard [mailto:BeckhamRE@cdm.com]  
Sent: Wednesday, July 16, 2003 5:30 PM  
To: 'RANEY@VOLPE.DOT.GOV'; Autio, Anni  
Subject: MOD Status

For MODs 27 and 28, I have email approvals from EPA, but have not been able to locate approvals from Volpe. CDM received a hardcopy of 27 with an original signature from RESI, that was subsequently forwarded to Volpe on 7/8/3. (Did I miss an approval email?) To my knowledge, a hardcopy of 28 has not been prepared.

- Richard Beckham

-----Original Message-----

From: Raney, Mark  
Sent: Wednesday, June 18, 2003 10:56 AM  
To: 'Mahoney, Ron'  
Cc: 'Anni Autio'; 'Mary Goldade'  
Subject: RE: EPA Markups: MOD LB-000025

Ron,

I concur with Mary's comments below. I provide Volpe's approval for MOD LB-000025 with Mary's changes and the addition of an estimate of the number of samples involved (i.e., < 20).

Thanks,

Mark.

-----Original Message-----

From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]  
Sent: Wednesday, June 04, 2003 9:27 AM  
To: 'Mark Raney'

7/18/2003

Cc: 'Anni Autio'; 'Mary Goldade'; CDM STAFF  
Subject: FW: EPA Markups: MOD LB-000025

Mark,

Do you have any other comments for this mod? Mary asked for an estimate of the number of samples involved, and we agreed on < 20. The number is more likely < 10, but we've decided to err on the conservative side.

If I can get your input, we can put this one to bed.

R.K. Mahoney  
Senior Analyst  
Special Projects Coordinator  
EMSL Analytical, Inc.  
Westmont, NJ  
800.220.3675, x1218  
[rmahoney@emsl.com](mailto:rmahoney@emsl.com)

-----Original Message-----

From: Mary Goldade [<mailto:mgoldade@peakpeak.com>]  
Sent: Wednesday, May 14, 2003 6:32 PM  
To: Beckham, Richard; 'Charlie LaCerra'; [jeanneorr@resienv.com](mailto:jeanneorr@resienv.com); [rdemalo@emsl.com](mailto:rdemalo@emsl.com); [rmahoney@emsl.com](mailto:rmahoney@emsl.com); 'William Longo'; [rhatfield@mastest.com](mailto:rhatfield@mastest.com); 'Bill Egeland'; [Bob.Shumate@battaenv.com](mailto:Bob.Shumate@battaenv.com); 'Naresh C. Batta'; 'Shu-Chun Su'; [corbin77@atc-enviro.com](mailto:corbin77@atc-enviro.com); 'Gustavo Delgado'; 'Garth B. Freeman'; Autio, Anni; [Raney@volpe.dot.gov](mailto:Raney@volpe.dot.gov); [brattin@syrres.com](mailto:brattin@syrres.com); [Goldade.mary@EPAMail.epa.gov](mailto:Goldade.mary@EPAMail.epa.gov); [dmazzaferro@mastest.com](mailto:dmazzaferro@mastest.com); [m\\_szynskie@resienv.com](mailto:m_szynskie@resienv.com)  
Subject: EPA Markups: MOD LB-000025

Suggested changes to the MOD are attached.  
Ron-Do you already have in hand an estimate regarding the actual number of samples this affects (i.e., are you able to quantify the term "few/limited"?)  
Thanks,  
Mary

----- Original Message -----

From: "Beckham, Richard" <[BeckhamRE@cdm.com](mailto:BeckhamRE@cdm.com)>  
To: "Charlie LaCerra" <[clacerra@emsl.com](mailto:clacerra@emsl.com)>; <[jeanneorr@resienv.com](mailto:jeanneorr@resienv.com)>; <[rdemalo@emsl.com](mailto:rdemalo@emsl.com)>; <[rmahoney@emsl.com](mailto:rmahoney@emsl.com)>; "William Longo" <[wlongo@mastest.com](mailto:wlongo@mastest.com)>; <[rhatfield@mastest.com](mailto:rhatfield@mastest.com)>; "Bill Egeland" <[begeland@mastest.com](mailto:begeland@mastest.com)>; <[Bob.Shumate@battaenv.com](mailto:Bob.Shumate@battaenv.com)>; "Naresh C. Batta" <[ncbatta@battaenv.com](mailto:ncbatta@battaenv.com)>; "Shu-Chun Su" <[scsu@delanet.com](mailto:scsu@delanet.com)>;

7/18/2003

<corbin77@atc-enviro.com>; "Gustavo Delgado"  
<gdelgado77@atc-enviro.com>;  
"Garth B. Freeman" <gfreeman@mastest.com>; "Autio, Anni"  
<AutioAH@cdm.com>; <Raney@volpe.dot.gov>; <brattin@syrres.com>;  
<Goldade.mary@EPAMail.epa.gov>; <dmazzaferro@mastest.com>;  
<mgoldade@peakpeak.com>; <m\_szynskie@resienv.com>  
Sent: Wednesday, May 14, 2003 3:28 PM  
Subject: MOD LB-000025

> This MOD impacts only EMSL. For your review and comment:  
>  
> <<LB-000025.doc>>  
> - Richard Beckham

<<LB-000025\_rev (MG 6-04-03 email).doc>> <<LB-000027 (MG  
6-24-03).doc>> <<LB-000028 (MG 6-24-03).doc>>  
>  
>  
>

7/18/2003



**Mary Goldade**

07/29/03 01:57 PM

To: Anni Autio

cc: Mark Raney

cc:

Subject: LB-000027 & LB-000028 are signed and mailed

Anni & Joe,

I have mail you the original copiew of the mods LB-000027 & LB-000028.

Several of the email approval pages were not provided. I attached them.

---

**Mary Goldade**

**Regional Superfund Chemist**



U.S. Environmental Protection Agency, Region 8

999 19<sup>th</sup> Street, Suite 300

Mail Code: BEPR-PS

Denver, CO 80202

Phone: (303) 312-7024

Fax: (303) 312-6065

email: goldade.mary@epa.gov



Request for Modification  
to  
Laboratory Activities  
LB-000029b

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.  
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs  
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): ☒ TEM-AHERA ☒ TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002  
EPA/600/R-93/116 ☒ ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03  
Other: \_\_\_\_\_

Requester: Lynn Woodbury Title: Technical consultant  
Company: Syracuse Research Corporation Date: December 7, 2006

Description of Modification:

Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses of air and dust. The general concepts presented in this modification may also be used for soil and water, but specific details regarding the frequency and interpretation of laboratory QC samples will need to be adjusted for these media.

Reason for Modification:

This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria.

Potential Implications of this Modification:

There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): ☒ All Individual(s) \_\_\_\_\_

Duration of Modification (circle one):

Temporary Date(s): \_\_\_\_\_  
Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

☒ Permanent (Complete Proposed Modification Section) Effective Date: \_\_\_\_\_  
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

☒ Not Applicable ☐ Reject ☐ Low Bias ☐ Estimate ☐ High Bias ☐ No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): \_\_\_\_\_

Technical Review: \_\_\_\_\_ Date: \_\_\_\_\_  
(Laboratory Manager or designate)

Project Review and Approval: \_\_\_\_\_ Date: 4/25/07  
(Volpe/Project Technical Lead or designate)

Approved By: Mary Goldack Date: 4/25/07  
(USEPA/Project Chemist or designate)

## DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

**Low Bias** - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

**High Bias** - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

## ***QC Sample Type Definitions***

There are three categories of TEM laboratory QC samples: Blanks, Recounts, and Repreparations.

### **Blanks**

Lab Blank (LB) – This is a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

### **Recounts**

Recount Same (RS) – This is a TEM grid that is re-examined within the same laboratory and by the same microscopist who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Same TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Recount Different (RD) – This is a TEM grid that is re-examined within the same laboratory but by a different microscopist than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Different TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Interlab (IL) - This is a TEM grid that is re-examined by a microscopist from a different laboratory than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Interlab TEM analyses for air and dust will be selected in accord with the procedure presented in Attachment 2.

Verified Analysis (VA) – This is a recount of a TEM grid (same grid openings) performed in accord with the protocol for verified analysis as provided in NIST (1994) (provided as Attachment 3). Verified TEM analyses will be selected in accord with the procedure presented in Attachment 1.

### **Repreparations**

Repreparation (RP) – This is a TEM grid that is prepared from a new portion of the same filter that was used to prepare the original grid. Typically this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter. Repreparations will be selected in accord with the procedure presented in Attachment 1.

## ***Frequency***

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Verified analysis	1%
Repreparation	1%
Interlab	0.5%
<b>Total</b>	<b>10%</b>

Each laboratory should prepare and analyze lab blank, recount (same, different and verified), and reparation samples at the minimum frequency specified in the table above. The selection procedure and laboratory SOP for the selection of samples for the purposes of recounts and reparation are provided in Attachment 1. Samples for interlab comparisons will be selected by EPA's technical consultant (SRC) in accord with the selection procedure and laboratory SOP provided in Attachment 2.

### ***Procedure for Evaluating QC Samples and Responses to Exceptions***

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: The procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

#### **Lab Blanks.**

There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

#### **Recounts.**

All recount samples (same, different, verified, and interlab) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. All structure enumeration and measurements will adhere to the established project-specific documentation presented in LB-000016A and LB-000031A. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 3).

Measurement parameter	Concordance Rule
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent)  For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent).  For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more areas of discordance, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Laboratory Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

#### Repreparations.

Repreparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 4. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

#### Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

QC Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with $\geq 1$ asbestos structures	0% - 0.1%	0.2% - 0.5%	>0.5%
Recounts	Concordance on LA count	>95%	85-95%	<85%
	Concordance on type (chrysotile vs. amphibole)	>99%	95%-99%	<95%
	Concordance on LA length	>90%	80%-90%	<80%
	Concordance on LA width	>90%	80%-90%	<80%
Repreps	Concordance on LA concentration/loading	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

#### REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

## ATTACHMENT 1

### Selection Procedure and Laboratory SOP for Recounts (RS, RD, VA) and Repreparations (RP)

#### Selection Procedure

As specified in the Frequency section above, the frequency of Recount Same (RS) should be 1%, the frequency of Recount Different (RD) should be 2.5%, the frequency of Verified Analyses (VA) should be 1%, and the frequency of Repreparations (RP) should be 1%, corresponding to a total within-laboratory QC frequency of 5.5% for these analysis types. This is approximately 1 QC sample per 20 field samples. Based on this frequency, it is possible to determine which laboratory job(s) will have one or more samples selected for recount analysis or reparation.

For those laboratory jobs in which a recount or reparation sample is to be selected, the analyst should record the total number of structures observed in each sample. The sample(s) selected for recount or reparation should be those within the laboratory job with the highest number of structures per grid opening (GO) area examined (calculated as the number of GOs evaluated \* the GO area). When selecting samples for reparation, if possible, preferentially select samples in which the total number of GOs is 40 or less. Because reparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, reparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the selection of samples with 40 GOs or less will reduce analytical costs associated with reparations. When selecting samples for recount, it is not necessary to impose a minimum or maximum number of GOs because concordance is evaluated on a GO and structure basis, rather than a concentration basis. If all samples within the laboratory job are non-detect, a non-detect sample may be selected. A non-detect sample should be preferentially selected, every 10<sup>th</sup> selection.

This selection procedure will ensure that the recount analyses and reparations yield a dataset best suited to assess concordance<sup>1</sup>.

#### Laboratory SOP for Recount Analyses

1. For recount samples, re-analyze the selected sample in accord with the appropriate procedures for each type of recount (RS, RD, or VA). If more than 10 GOs were evaluated in the original analysis, the original analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the recount analysis. The original analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures per GO area examined, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated.
2. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Recount Same", "Recount Different", or "Verified Analysis", as appropriate. Be sure that the grid and GO names match exactly with the names evaluated in the original analysis (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the original analyst or laboratory director to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).
3. If there is one or more discordant GOs between the original analysis and the recount analysis, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

<sup>1</sup> It should be noted that this selection procedure will tend to result in the preferential selection of samples with the highest air concentration/dust loading values. Thus, summary statistics based on laboratory QC samples may tend to be biased high.

4. Submit the recount TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

#### Laboratory SOP for Repreparations

1. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.
2. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.
3. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".
4. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.



## ATTACHMENT 2

### Selection Procedure and Laboratory SOP for Interlabs (IL)

#### Selection Procedure

1. On the 1st of each month, EPA's technical consultant (SRC) will compile a list of all samples for which air and dust TEM results (ISO+AHERA+ASTM) were uploaded into Libby V2 Database in the preceding month (e.g., on November 1<sup>st</sup>, specify a date range of Oct 1-31, 2005). The Libby V2 Database query will be based on the upload date rather than the analysis date to ensure that analyses with an upload in a different month as the analysis date were not excluded<sup>2</sup>.
2. Identify the target number of air and dust interlab samples needed to meet the QC requirements for interlabs specified in the Frequency section above (0.5%). This is accomplished by multiplying the desired interlab frequency (0.5%) by the total number of air and dust analyses performed in the preceding month. For example, 178 TEM air analyses in October 2005 \* 0.5% = 0.89 (which is rounded up to 1). At a minimum, at least one air and one dust sample will be selected for interlab analysis.
3. For each medium (air and dust), rank order the TEM analyses from the preceding month on the total number of LA structures per GO area examined (calculated as the number of GOs evaluated \* the GO area). Selecting from analyses with a high number of LA structures per GO area examined increases the likelihood that the GOs evaluated as part of the interlab analysis will have one or more LA structures.
4. Exclude samples in which the total number of GOs is more than 40 GOs<sup>3</sup>. Exclude any samples that have already been selected for interlab evaluation previously.
5. Select the appropriate number of air and dust interlab samples from the available TEM analyses for which the total number of LA structures per GO area examined is higher than 0 (i.e., LA detects). If the total number of samples with LA detects is equal to the desired number of interlab samples, select all detected samples for interlab analysis. If the total number of samples with LA detects is less than the desired number of interlab samples, select non-detect samples for interlab analysis. If the total number of samples with LA detects is higher to the desired number of samples, interlab samples will be selected to represent multiple laboratories, selecting those samples with the highest number of LA structures per GO examined first. EPA's technical consultant (SRC) will keep a running total of the number of samples selected by laboratory to ensure that the long-term frequency of interlabs for each laboratory is generally similar.
6. Submit list of selected interlab samples to the CDM Laboratory Manager.
7. Each month, the CDM Laboratory Manager will provide each laboratory with the list of samples selected for Interlab analysis.

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<sup>2</sup> Consider the case where the TEM analysis for sample X-12345 was performed on September 22 and the results were uploaded on October 3. The interlab selection query performed on October 1, if limited to all results analyzed from September 1-30, would not capture the results for X-12345 because they had not yet been uploaded. The interlab selection query performed on November 1, limited to all results analyzed from October 1-31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

<sup>3</sup> Because all interlabs will be reprepared, these interlab reparation samples will also be evaluated for concordance with the original sample. Because reparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, reparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the focusing on samples with 40 GOs or less will reduce analytical costs associated with reparations.

## Laboratory SOP

### **At the Originating Laboratory:**

1. Upon receipt of the interlab sample list from the CDM Laboratory Manager, locate the appropriate sample filter. If less than ¼ of the sample filter is available, contact the CDM Laboratory Manager to identify an interlab replacement sample.
2. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.
3. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the re-preparation. Place the remaining grid in storage.
4. Record the orientation of each grid using the instructions for grid orientation specified in NVLAP (see Attachment 5).
5. When performing the TEM analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.
6. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".
7. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.
8. Identify which laboratory will perform the interlab analysis in accord with the following table:

Originating Lab	Lab for Interlab Sample #1	Lab for Interlab Sample #2	Lab for Interlab Sample #3	Lab for Interlab Sample #4	Lab for Interlab Sample #5	Lab for Interlab Sample #6...
Hygeia	Batta	MAS	RESI	EMSL-L	EMSL-W	Repeat... (beginning with the Lab identified for Sample #1)
Batta	MAS	RESI	EMSL-L	EMSL-W	Hygeia	
MAS	RESI	EMSL-L	EMSL-W	Hygeia	Batta	
RESI	EMSL-L	EMSL-W	Hygeia	Batta	MAS	
EMSL-L	EMSL-W	Hygeia	Batta	MAS	RESI	
EMSL-W	Hygeia	Batta	MAS	RESI	EMSL-L	

EMSL-L = EMSL, Mobile Lab in Libby

EMSL-W = EMSL, Westmont

9. If more than 10 GOs were evaluated in the repreparation analysis, the repreparation analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the interlab analysis. The repreparation analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures, which may be analyzed in the event that the selected GO is damaged and cannot be re-evaluated.
10. Ship the grid(s) for the interlab sample to the appropriate laboratory using standard chain of custody procedures. For each interlab sample, include a list of which GOs should be evaluated for each grid. The names of the grid and GOs provided on the chain of custody form should match exactly with those recorded in the original TEM data recording spreadsheet (including dashes, underscores, and spaces).
11. After the interlab laboratory has completed the interlab analysis, it will request copies of the hard copy laboratory benchsheet(s), the grid opening sketches, and TEM file for each interlab sample.

12. If areas of discordance are noted, the senior laboratory analyst from the interlab laboratory will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

**At the Interlab Laboratory:**

1. For each grid provided for interlab analysis, place the grid into the TEM grid holder ensuring that the grid orientation matches that which was specified by the originating laboratory (see Attachment 5 for details).
2. For the 10 GOs identified for interlab analysis, perform TEM analysis using the analysis method and counting rules specified on the chain of custody. Be sure that the grid and GO names match exactly with the names provided on the chain of custody (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the originating laboratory to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).
3. When performing the TEM interlab analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.
4. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Interlab".
5. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.
6. Contact the originating laboratory to request copies of the hard copy laboratory benchsheet(s), grid opening sketches, and TEM file for each interlab sample.
7. Perform a verified analysis using the procedures presented in NIST (1994) (provided as Attachment 3).
8. Assess the between-laboratory concordance, both on a GO-by-GO basis and on a structure-by-structure basis, using the Libby-specific recount concordance rules. If areas of discordance are noted, the senior laboratory analyst will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).
9. Summarize the results of the verified analysis and document any changes in laboratory procedures or analyst training that were implemented to address noted discordances. Provide a copy of this report to EPA Chemist and the CDM Laboratory Manager.
10. Ship the grid(s) back to the originating lab.

## **ATTACHMENT 3**

**Airborne Asbestos Method:  
Standard Test Method for Verified Analysis of Asbestos  
by Transmission Electron Microscopy-Version 2.0.  
NIST (1994)**

**NISTIR 5351**

# **Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0**

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March 1994



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## Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

## 1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

## 2. Terminology

### 2.1 Definitions:

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

### 2.2 Description of Terms Specific to This Standard:

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos-containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*<sup>1</sup>--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*<sup>2</sup>--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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<sup>1</sup>Code Fed. Reg. 1987, 52 (No. 210), 41826-41905.

<sup>2</sup>ISO 10312 1993, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*--the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*--structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true positive-unmatched)*--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*--structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories--type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*--reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.



### 3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted vs time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values  $\geq 0.85$  and FP/TNS values  $\leq 0.05$ . The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

### 4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2-- The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3-- The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4-- The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight forward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5-- The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

## 5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator 1}) + TPU(\text{Operator 2}) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$I = TP/TNS + FN/TNS$$

## 6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

## 7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

## APPENDIXES

(Nonmandatory Information)

## X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: \_\_\_\_\_

Date: \_\_\_\_\_

Grid slot: \_\_\_\_\_

Verifying Analyst: \_\_\_\_\_

Grid square: \_\_\_\_\_

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

\*The values for these items will be the same for both analyses.

## Test Report Form (continued)




1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

## X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

Analyst 2








Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2





Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.



Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

Analyst 2



Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1		FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1		FP1	1	Chr

Analyst 2



Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

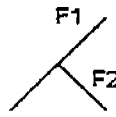

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
					
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

## Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

## Analyst 2

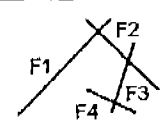
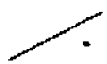

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.


## Analyst 1


Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

## Analyst 2

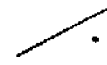
Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr


a

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

b




Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr


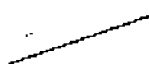
c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1			1	Chr
1.0	0.1		TPM2	1	Chr




Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr



a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).




Analyst 1



Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr

b


Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

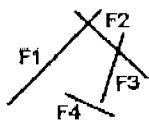
c

Fig. X2.8 (caption on previous page).

## Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
5	3			1	Chr

## Analyst 2


Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
					
5	0.1	F1		1	Chr
3	0.1	F2		1	Chr
2	0.1	F3		1	Chr
1	0.1	F4		1	Chr

a

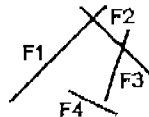
Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).



## Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

## Analyst 2

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

b

Fig. X2.9 (caption on previous page)

### X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

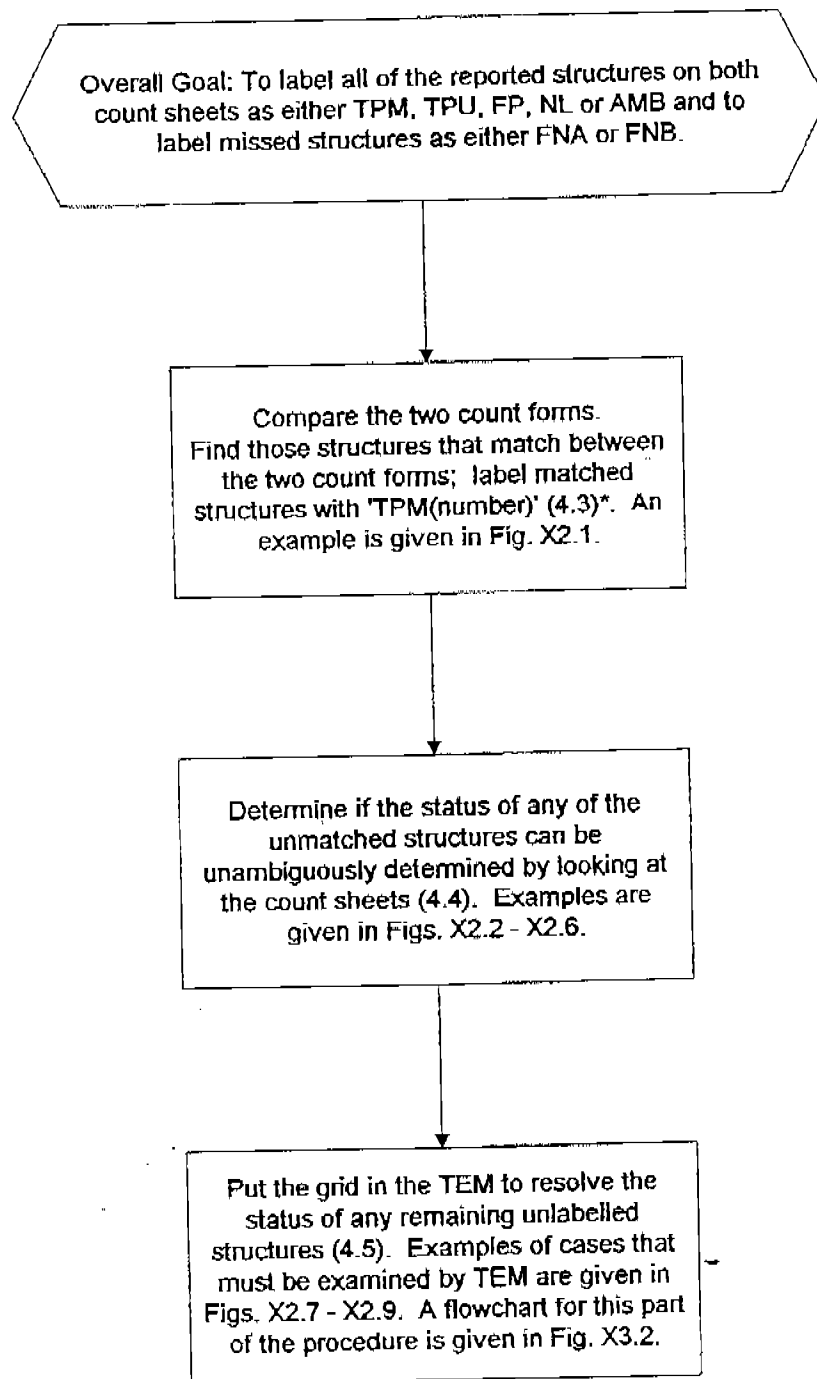


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst.  
\*Numbers in parentheses in each block refer to the item number in the procedure.

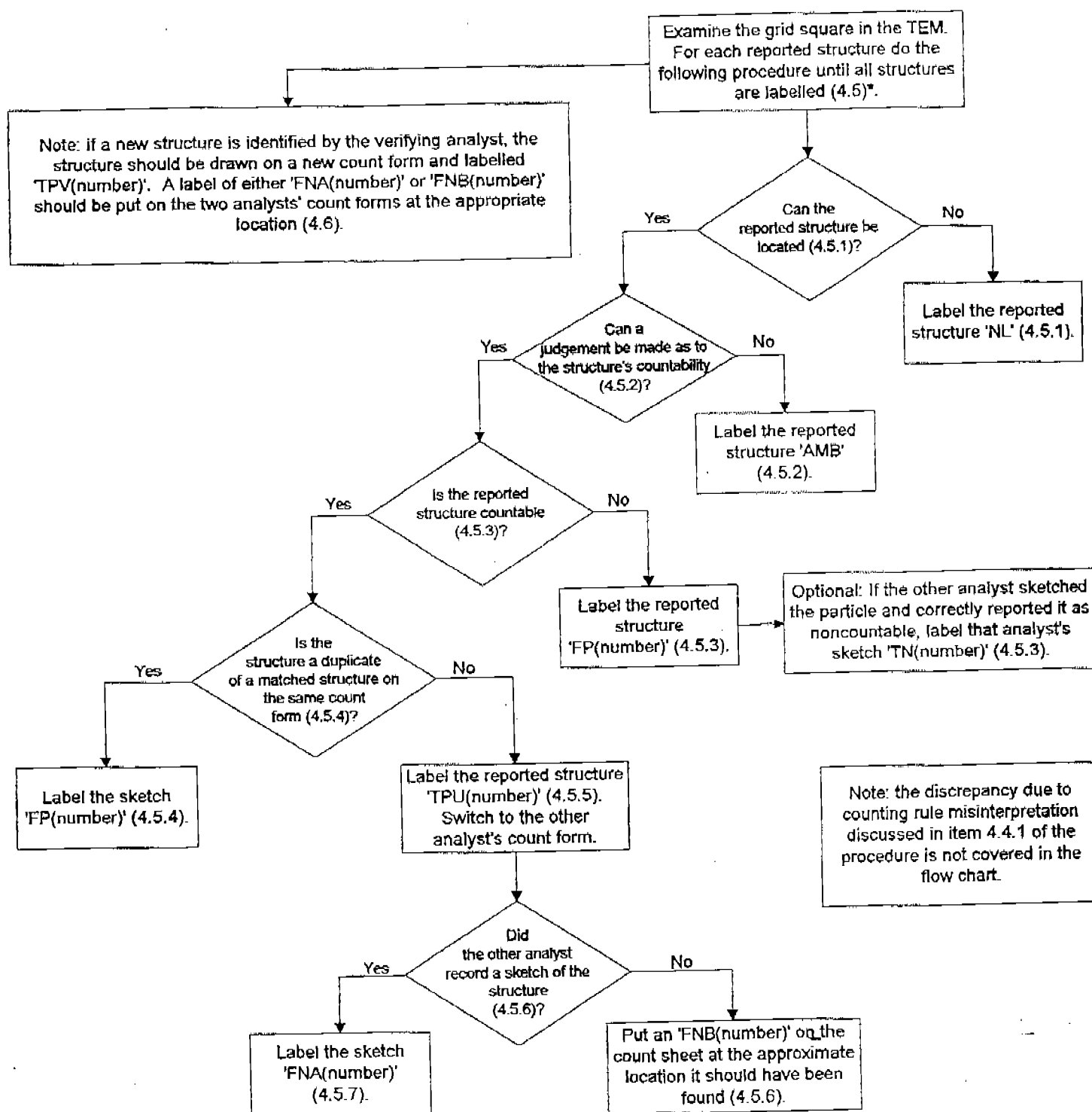


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. \*Numbers in parentheses in each block refer to the item number in the procedure.

## ATTACHMENT 4

### Statistical Comparison of Two Poisson Rates

#### 1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the reparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from reparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

#### 2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation  
S1 = Sensitivity of first evaluation  
N2 = Fiber count in second evaluation  
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1  
Rate 2 = N2 · S2  
Ratio = Rate 1 / Rate 2

$$\text{Lower Bound} = \left( \frac{S1}{S2} \right) \left( \frac{N1}{N2 + 1} \right) / F \left[ \frac{1 + \gamma}{2}; 2 \cdot N2 + 2, 2 \cdot N1 \right]$$
$$\text{Upper Bound} = \left( \frac{S1}{S2} \right) \left( \frac{N1 + 1}{N2} \right) \cdot F \left[ \frac{1 + \gamma}{2}; 2 \cdot N1 + 2, 2 \cdot N2 \right]$$

where  $\gamma$  is the confidence interval (e.g., 0.95) and  $F[\delta; df1, df2]$  is the 100 $\delta$ th percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is  $> 1$ , then it concluded that rate 1 is greater than rate 2 at the 100(1- $\gamma$ )% significance level. If the upper bound of the ratio is  $< 1$ , then it concluded that rate 1 is less than rate 2 at the 100(1- $\gamma$ )% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1- $\gamma$ )% significance level.

#### Example:

N1 = 4 structures  
S1 = 0.0001 (cc)<sup>-1</sup>  
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures  
S2 = 0.001 (cc)<sup>-1</sup>  
Rate 2 = 6 · 0.001 = 0.006 s/cc

$\gamma = 0.95$

$$Lower\ Bound = \left( \frac{0.0001}{0.001} \right) \left( \frac{4}{6+1} \right) / F \left[ \frac{1+0.95}{2}; 2 \cdot 6 + 2, 2 \cdot 4 \right] = 0.014$$

$$Upper\ Bound = \left( \frac{0.0001}{0.001} \right) \left( \frac{4+1}{6} \right) \cdot F \left[ \frac{1+0.95}{2}; 2 \cdot 4 + 2, 2 \cdot 6 \right] = 0.281$$

In this example, because the upper bound of the ratio is < 1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

### 3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

## **ATTACHMENT 5**

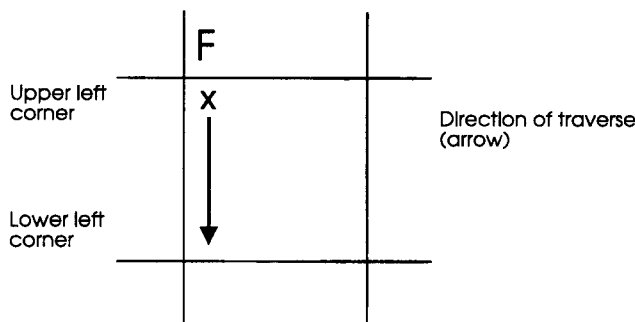
### **NVLAP Airborne Asbestos Proficiency Test 98-2: Grid Orientation**

## NVLAP AIRBORNE ASBESTOS PROFICIENCY TEST 98-2

### *Instructions for Form 1*

The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on *Form 1*. Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. ***Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted.*** See the example on *Form 1*.
2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.
3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1*. Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).
4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



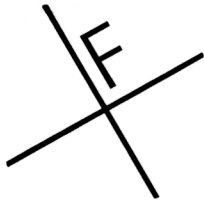
The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

NVLAP Lab Code: \_\_\_\_\_

**Form 1. Grid Orientation**

1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

EXAMPLE:



2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

EXAMPLE:





## **ATTACHMENT 6**

### **Grid Opening Template for Sketching the Relative Position of Observed Structures**

## STRUCTURE LOCATIONS WITHIN GRID OPENING

**\*\*\*NOTE:** *Sketches only need to be completed for interlab analyses and reprep associated with interlabs*

Lab Name: \_\_\_\_\_ Lab Job Number: \_\_\_\_\_

Index ID: \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Lab QC Type (circle one):      Reprep for interlab      Interlab

Grid: \_\_\_\_\_ Grid Opening: \_\_\_\_\_

upper  
left  
corner

traverse direction

Comments:



**Request for Modification**  
To  
**Laboratory Activities**  
LB-000030

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**

**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin

Title: Technical consultant

Company: Syracuse Research Corporation

Date: 5 August 2003

**Description of Modification:**

All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance and orientation relative to any nearby landmarks, if present.

morphology,

**Reason for Modification:**

This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis.

**Potential Implications of this Modification:**

There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): All

Individual: \_\_\_\_\_

**Duration of Modification (circle one):**

Temporary

Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent

(complete Proposed Modification Section)

Effective Date: 8/14/03 (insert based on date of final approval)

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

**Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):**

Technical Review: WJ Brattin  
(Laboratory Manager or designate)

Date: 8/14/03

Project Review and Approval: [Signature]  
(Volpe: Project Technical Lead or designate)

Date: 8/19/03

Approved By: [Signature]  
(USEPA: Project Chemist or designate)

Date: 8/14/03

## Autio, Anni

---

**From:** Goldade.Mary@epamail.epa.gov  
**Sent:** Thursday, August 07, 2003 10:43 AM  
**To:** Autio, Anni  
**Cc:** Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo  
**Subject:** EPA Comments: LB-000030 (Draft for review/comment)



LB-000030 v0 (MG pic08313.gif (3 KB)  
08-07-03).doc...

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)



## Request for Modification

To  
Laboratory Activities  
LB-000030

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**

**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin Title: Technical consultant  
Company: Syracuse Research Corporation Date: 5 August 2003

### Description of Modification:

All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology and orientation relative to any nearby landmarks, if present.

Deleted: i

### Reason for Modification:

This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis.

### Potential Implications of this Modification:

There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis.

Laboratory Applicability (circle one): All Individual: \_\_\_\_\_

### Duration of Modification (circle one):

Temporary Date(s): \_\_\_\_\_  
Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (complete Proposed Modification Section) Effective Date: (insert based on date of final approval)

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Deleted:

Technical Review: \_\_\_\_\_ Date: \_\_\_\_\_  
(Laboratory Manager or designate)

Project Review and Approval: \_\_\_\_\_ Date: \_\_\_\_\_  
(Volpe: Project Technical Lead or designate)

Approved By: \_\_\_\_\_ Date: \_\_\_\_\_  
(USEPA: Project Chemist or designate)

## Autio, Anni

---

**From:** DeMalo, Robert [RDemalo@EMSL.com]  
**Sent:** Thursday, August 07, 2003 11:20 AM  
**To:** Goldade.Mary@epamail.epa.gov; Autio, Anni  
**Cc:** Bob Shumate; LaCerra, Charles; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; EMSL Mobile Lab - Asbestos; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Richard Hatfield; Mahoney, Ron; Shu-Chun Su; Bill Longo  
**Subject:** RE: EPA Comments: LB-000030 (Draft for review/comment)

I propose adding the word "morphology" as well into the description, as noted. I have no problem with including ISO to this procedure.

-----Original Message-----

**From:** Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]  
**Sent:** Thursday, August 07, 2003 10:43 AM  
**To:** Autio, Anni  
**Cc:** Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo  
**Subject:** EPA Comments: LB-000030 (Draft for review/comment)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)

**Autio, Anni**

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**From:** Raney, Mark [RANEY@VOLPE.DOT.GOV]  
**Sent:** Thursday, August 14, 2003 10:41 AM  
**To:** 'Goldade.Mary@epamail.epa.gov'; Autio, Anni  
**Cc:** Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Raney, Mark; Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo  
**Subject:** RE: EPA Comments: LB-000030 (Draft for review/comment)



LB-000030 v0 (MR  
08-14-03).doc...

I concur with Mary's recommendations and mark-ups. The attached version also includes Rob Demalo's recommendation of adding morphology under the description section. Bill please finalize, sign and send it through the signature process. To expedite the process could you get Mary to sign before providing the original on for my signature. Let me know if you have any questions.

Thanks,

Mark.

-----Original Message-----

**From:** Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]  
**Sent:** Thursday, August 07, 2003 10:43 AM  
**To:** Autio, Anni  
**Cc:** Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo  
**Subject:** EPA Comments: LB-000030 (Draft for review/comment)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)



## Request for Modification

To  
Laboratory Activities  
LB-000030

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.  
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin Title: Technical consultant  
Company: Syracuse Research Corporation Date: 5 August 2003

### Description of Modification:

All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology and orientation relative to any nearby landmarks, if present.

Deleted: i

### Reason for Modification:

This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis.

### Potential Implications of this Modification:

There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis.

Laboratory Applicability (circle one): All Individual: \_\_\_\_\_

### Duration of Modification (circle one):

Temporary Date(s): \_\_\_\_\_  
Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (complete Proposed Modification Section) Effective Date: (insert based on date of final approval)

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Deleted:

Technical Review: \_\_\_\_\_ Date: \_\_\_\_\_  
(Laboratory Manager or designate)

Project Review and Approval: \_\_\_\_\_ Date: \_\_\_\_\_  
(Volpe: Project Technical Lead or designate)

Approved By: \_\_\_\_\_ Date: \_\_\_\_\_  
(USEPA: Project Chemist or designate)





**Request for Modification**  
to  
**Laboratory Activities**  
LB-000066c

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**

**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002  
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03  
Other: \_\_\_\_\_

Requester: W. Brattin Title: Technical Consultant  
Company: Syracuse Research Corporation Date: 09/11/2007

**Description of Modification:**

This temporary modification applies to all investigative samples (as defined by the most recent version of LB-000053) evaluated at the Libby Superfund site. Based on this temporary modification, all analytical laboratories shall: 1) begin to utilize the structure comment field to further characterize particles with regard to the levels (presence/absence) of the sodium and potassium peaks observed in the EDS spectrum; 2) record on the data sheets all NAM particles that are "close calls" (defined in attachment 1); 3) increase the frequency that EDS spectra are saved for "LA" and "close call" structures; 4) increase the frequency that photographic images of particle morphology are recorded for "LA" and "close call" structures, and 5) utilize the comment field to record mineral type of each recorded particle, including LA, OA, C and "close call" NAM particles.

**Reason for Modification:**

Studies of asbestos from the mine in Libby indicate that the asbestos spans several different mineralogical classes, including winchite and richterite (these are the primary forms) as well as tremolite and possibly actinolite (these are minor forms) (Meeker et al, 2003). Consequently, all analytical laboratories supporting the Libby project are currently directed to classify as "LA" any particle in an investigative sample that a) meets morphological requirements (e.g., length  $\geq 0.5$   $\mu$ m, aspect ratio  $\geq 3:1$ ), b) has an SAED diffraction pattern that is consistent with amphibole, and c) has an EDS spectrum that is consistent with the range of mineral forms observed in the mine in Libby (USEPA 2005). To date, this method for designating "LA" to a particle has worked well for samples collected at the Libby Site. However, a recent project that included collection of air samples from locations outside of Libby highlighted a potential limitation of this approach. That is, tremolite and actinolite are included in the "LA" suite and are found in Libby, but these types of fibers may also occur as the result of releases from sources that are not related to the mine in Libby (e.g., commercial products or natural sources). Also, some other minerals (e.g., pyroxenes) are sometimes difficult to distinguish from actinolite and tremolite (Bern et al. 2002). Because mineralogical data may or may not inform our understanding of the toxicity of LA, delineating amongst these mineral types is desirable at this stage of data collection. Therefore, the primary focus of this temporary modification is to collect more detailed data on the frequency of occurrence of sodium and potassium-containing particles both for samples from Libby and for samples from other locations.

**Potential Implications of this Modification:**

This temporary modification does not change any current procedures other than to require more detailed recording of data on particles observed under TEM. These additional requirements are not associated with a significant increase in time or cost of analysis. Hence, there are no negative implications of the modification.



Laboratory Applicability (circle one): ☒ All Individual(s) \_\_\_\_\_

Duration of Modification (circle one):

☒ Temporary Date(s): 09/12/2007 until notified

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: \_\_\_\_\_

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

☒ Not Applicable

☐ Reject

☐ Low Bias

☐ Estimate

☐ High Bias

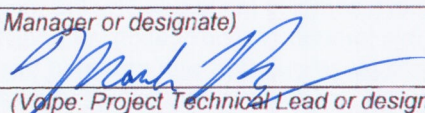
☐ No Bias

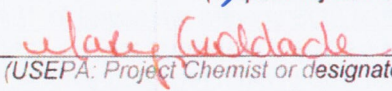
Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

See Attachment 1

Note: This modification (LB-000066c) **supersedes** LB-000066b.

Technical Review: \_\_\_\_\_ Date: \_\_\_\_\_  
(Laboratory Manager or designate)

Project Review and Approval:  Date: 9/12/07  
(Volpe: Project Technical Lead or designate)

Approved By:  Date: 9/11/07  
(USEPA: Project Chemist or designate)

## REFERENCES

Bern A, Meeker G, Brownfield I. 2002. Guide to Analysis of Soil samples from Libby, Montana for Asbestos Content by Scanning Electron Microscope and Energy Dispersive Spectroscopy. U. S. Geological Survey Administrative Report. October 17, 2002.

Meeker GP, Bern AM, Brownfield IK, Lowers HA, Sutley SJ, Hoeffen TM, and Vance JS. 2003. The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby Montana. American Mineralogist 88:1955-1969.

USEPA. 2005. EDS Spectra Characteristic Study for Libby-Type Amphiboles. Report prepared by Syracuse Research Corporation, Denver CO, for USEPA, Region 8, Denver CO. March 15, 2005.

## DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

**Low Bias** - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

**High Bias** - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.



## ATTACHMENT 1

1. Continue to classify structures as LA, OA, or C in accord with current procedures.
2. For all NAM particles that were "close calls" (i.e., they required careful assessment to determine they were not LA or OA), record the NAM particle on the bench sheet. Be sure to place a zero in the "total" column to ensure the particle is not counted as an asbestos fiber. NAM particles such as vermiculite, biotite, hydrobiotite, gypsum, titanium and other minerals that are clearly not amphibole should not be recorded.
3. For all particles that are recorded (including NAMs), use the structure comment field to record one of the following comments:

Code	Meaning
NaK	Na and K are both clearly present
NaX	Only Na is clearly present
XK	Only K is clearly present
XX	Na and K are not clearly present

4. For all particles that are recorded, whenever possible, use the structure comment field to identify a probable mineral classification. Use the designation "WRTA" (winchite/richterite/tremolite/actinolite) to indicate a particle that is consistent in morphology and chemical composition with a particle that is likely to have originated from the vermiculite mine in Libby. This will include most NaK particles and may include some NaX and some XK particles. It is unlikely that this will include any XX particles. For all other particles, use the following codes:

AC – actinolite  
TR – tremolite  
AT – actinolite/tremolite (too close to call)  
AM – amosite  
AN – anthophyllite  
CR – crocidolite  
PY – pyroxene  
UN – Unknown

5. Increase the frequency that EDS spectra are recorded (saved). For each sample, record the EDS for each LA and each "close call" particle, up to a maximum of 5 LA and 5 "close call" particles per sample. To the extent practical, collect the EDS spectrum for a sufficient length of time that key peaks (e.g., sodium, potassium, aluminum), if present, can be clearly distinguished from background. Be sure that each EDS spectrum that is recorded can be linked to a specific particle in the EDD.
6. Increase the frequency that photomicrographic images of particle morphology are collected. For each particle for which an EDS spectrum is collected (up to 5 LA and 5 "close call" NAM, as discussed above), also record a photomicrograph of the same structures. Use the structure-specific comment field to record the photo identification number of each structure that is photographed. Convert all photographs to high quality electronic images (e.g., by scanning), and transmit the photos to CDM for evaluation.
7. Figure 1 provides a flow chart that summarizes the process implemented by this temporary modification.

**FIGURE 1**  
**FLOW CHART SUMMARIZING THIS TEMPORARY MODIFICATION**

